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CONTENTS OF THIS ISSUE

Contents of previous issues can be found by consulting the Industrial Arts Index at your library

General Index

The Nature of Cellulose: An Historical Review..... 55
By E. Heuser
Obituary 77
Book Reviews 77
Washington News Letter..... 78
By Waldon Fawcett
Open Forum 84

Index to Proceedings of
American Association of Textile Chemists and Colorists

Fifteenth Annual Meeting:
Fifteenth Annual Banquet P61
Remarks by E. D. Bass..... P62
Remarks by L. A. Olney..... P64
Remarks by Robert E. Rose..... P65
Saturday afternoon session:
Peroxide Bleach Baths and Some Factors Affecting Their Stability..... P67
A paper by D. J. Campbell
Synthetic Resins: Their Application to Textiles.. P71
A paper by D. H. Powers
Meeting, South Central Section..... P66
North Carolina State College Student Section..... P66
Meeting, Northern New England Section..... P70
Notice to Manufacturers and Importers of Chemicals for the Textile Industry..... P70
Annual Meeting, Piedmont Section..... P75
Delegates of Inter-Society Color Council..... P75
Applicants for Membership..... P75
Meeting, South Central Section..... P76
Calendar of Coming Events..... P76

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"To promote increase of knowledge of the application of dyes and chemicals in the Textile Industry.

"To encourage in any practicable ways research work on chemical processes and materials of importance to the Textile Industry.

"To establish for the members channels by which the interchange of professional knowledge among them may be increased."

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The Nature of Cellulose

*An Historical Review**

By E. HEUSER**

I. THE MOLECULAR STRUCTURE

An old French saying is "On retourne toujours à son premier amour." This somewhat freely applied to the chemical constitution of cellulose means that those of the investigators who were in love with the chain structure of cellulose, such as or similar to the well-known formula of Tollens, but had abandoned it for various reasons, have now returned to it. Ruefully, one may say, for the rather speculative period which about 15 years ago started a new era of cellulose research, although attractive in many ways, led us somewhat astray, while we should have maintained more scientific cold-bloodedness.

However, the indulgence in this speculation was no loss, since there was behind it a vast amount of experimental data, which, now seen in another light, can be utilized to support the modern concept of the cellulose structure. We have learned a great number of things, and, after all, it appears as if it was our destiny to go through these various phases in order to land so much surer on the solid ground on which I feel we now stand.

In order to arrive at a better understanding of our present conception on the chemical structure of cellulose it appears interesting enough to review the past history from the viewpoints indicated above.

At the time cellulose research received its new impulse, the following four facts were known:

1. The product of hydrolysis is glucose.
2. Acetolysis renders cellobiose.
3. There are probably not more than three hydroxyls which react as in alcoholic groups.
4. There is little indication of the existence of a free aldehyde group.

Since no other products of hydrolysis had been found, the first two facts justified the definition of cellulose as a polysaccharide built up from single mono- or di-saccharides. Trained in the classical school of tackling problems of chemical constitution it was only natural and logical for Tollens that he thought the glucose anhydrides as being linked together in accordance with the principle of polymerization in its original meaning, that is by condensation of glucose units under loss of water. Logical and natural also that he arranged the units in a chain. The amorphous nature of the cellulose, its insolubility and other properties placed it in the class of substances of high molecular weight. (*Fig.* 1.)

Tollens had established his hypothetical chain formula about 1895[1]. He also had a very distinct concept of the way in which the single units were linked together, that is, the carbonyl group of the one glucose being connected with Nos. 5 and 6 hydroxyl of the other—the so-called double oxygen linking with which he explains the particular physical properties of cellulose. Thus, the only thing for the following generation to do was to deliver experimental proof for Tollens' formula and not to bother too much about the high-molecular nature. This statement may cause my audience to call me a pedantical schoolmaster. This you may do. However much truth may be behind this statement, I would like you to accept it for the moment as a means of showing how the development would have proceeded if it had not been interrupted by the numerous excursions into the field of speculation about the nature of high-molecular substances.

Assuming that the hydrolysis of cellulose rendered 100 per cent glucose, so that this monose was the end product of the process, the problem was to lead the hydrolysis in such a way as to isolate the oligosaccharides whose possible formation Tollens' formula suggested. The more so, since the next higher link to glucose, the cellobiose, had been found already, and moreover, since it was known that nature produces tri- and tetrasaccharides and that some even had been synthesized at that time. Had the cellulose chemists succeeded in identifying the so-called "cellulose dextrines" as a mixture of cellotriose, cellotetrose, pentose, hexose, with still higher members, the road would have been opened to answer the next question,

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namely, as to how in these oligosaccharides the glucose units were linked together. For answering this question the actually isolated cellobiose would already have delivered an excellent material of investigation, the more so since the mode of linking between glucose molecules in other disaccharides was known to be of the glucosidic nature. The third question then would have been: Are the linkings in the cellulose itself always the same or do different linkings alternate?

Thus there were quite a number of problems open to tackle before the last questions called for an answer,

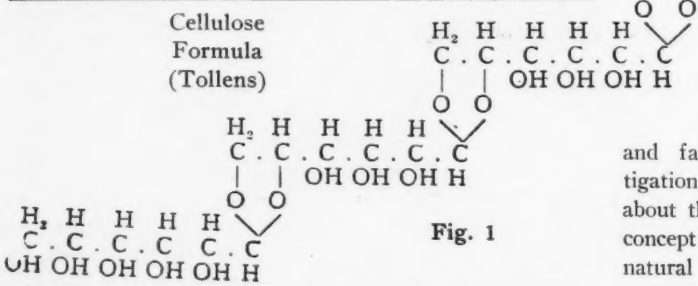


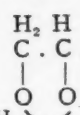
Fig. 1

namely, that of the length of the chain, i.e., how many glucose residues unite to form the cellulose molecule and finally how many molecules unite to form the micella.

Such probably would have been the way which structural chemistry would have taken if the generation of Tollens' had continued to interest themselves in this problem. However, for about 20 years very little happened except that a number of other formulae were proposed for which, however, no new facts were available. The only constitutionally important result may be seen in the more firmly established postulation that any cellulose formula must have only three esterifiable hydroxyls. This was the outcome particularly of Ost's indefatigable studies on the acetylation of cellulose. In reality the new formulae, as for instance that suggested by Vignon and later by Green, are only the result of a more or less unfounded speculation on the possible arrangement of the atoms in the glucose anhydride. Besides they do not explain the formation of cellobiose.

It is strange that such speculations were not replaced by experimentation at a time when Emil Fischer, the great experimenter, was leading the sugar chemistry and later that of the proteins through his school, which was so averse towards any speculation, from climax to climax. If we shall believe *K. Freundenberg*[2], Emil Fischer was of the opinion that cellulose and starch "were not yet ripe" for going into the nature of their chemical constitution. It appears as if this, his opinion, actually kept investigators away since it was only one or two years after his death, which occurred in 1919, that cellulose became the subject of new interest.

From that time on we find research to move chiefly in two directions. In the one it follows the solid and secure paths which for a long time had been established for the investigation of problems pertaining to the chemical constitution of matter and which had so successfully enlightened us on the chemical structure of many natural products. As for the other direction, research leaves the solid ground of approved principles and enters the realm of speculation on the size of the cellulose molecule trying to explain its nature as that of a high-molecular substance by means of a concept entirely new in the field of polysaccharide chemistry.



Roentgenography, although at its start nourishing such speculation, yet chiefly tried to bring its findings into accordance with the unshaken concept of chemical constitution, and the more evidence of its usefulness the latter produced the more did X-ray evaluation purge itself from premature and far too vague conclusions. Thus, X-ray investigation, although limited as to what it can tell us about the chemical side, has given much support to our concept of the chemical structure of cellulose and other natural products as we have it today.

Before research started to follow these two directions and when X-ray investigation had just produced its first findings on the crystalline structure of high molecular substances, a new formula was offered to the cellulose chemists in 1920 by *C. Hess*[3]. His formula, a penta-glucosido-glucose, based on the then prevailing ideas on the structure of tannic substances, represented a hexasaccharide in which five glucose units were linked, by means of glucoside linkings, with the five hydroxyls of another glucose unit. Although this formula was entirely of a speculative nature it maintains its importance in cellulose history for having awakened a number of investigators to activity and thus having ended the long and rather fruitless period, which lies between Tollens' formula and Emil Fischer's passing.

It was this point, that Hess' formula would account only for a yield of 32-33 per cent cellobiose and thus was not in agreement with the fact of a 40 per cent yield actually obtained, which caused *Karrer*[4] and *Freundenberg*[5], independently of each other, to study anew the acetolysis of cellulose.

Freundenberg[5] found that not only 40 but 61 per cent of cellobiose are formed and that these 61 per cent could be isolated if it were not for about 20 per cent which are lost again by destruction. A calculation based on a chain length of 100 glucose units revealed that the probably highest amount which could exist in the mixture after acetolysis is 67 per cent. While thus the penta-glucosido-glucose formula was not in agreement with one of the most important facts, the results of the acetolysis pointed in the direction of a long chain, i.e., the principle put forward in Tollens' suggestion.

At about the same time the method of total methylation and hydrolysis of the methylated products as applied by Irvine, by Haworth, and by Karrer and their collaborators,

though still at its beginning, already proved an excellent means of information on structural problems.

But the interesting results which thus (and somewhat later also from the use of phosphorpentabromide by Karrer) were produced on the probable structure of maltose, cellobiose, and other disaccharides[6], were overshadowed by the endeavors to find another explanation for the high-molecular state of natural products than previously had existed. These endeavors can be traced back to *Cross* and *Bevan*[7], the pioneers in the cellulose field, who expressed the opinion that cellulose may be an aggregation of units different from ordinary molecules, but the issue was started anew by the same formula, which brought those investigators to the fore, who did not see any necessity of changing the classical concept of chemical structure in the case of highly polymeric substances.

With his formula Hess had at the same time offered the suggestion that the cellulose molecule is represented by a number of hexasaccharide units kept together by means of "residual forces of affinity." This idea, possibly influenced by Werner's co-ordination theory on the nature of complex molecules, although or because being a deviation from the heretofore accepted meaning of polymerization, appeared for many a welcome possibility of widening their knowledge in this respect. From then on (it was the year 1920), the idea of the small units, held together by forces other than normal valencies, gains much headway.

With the penta-glucoside-glucose formula abandoned, the new idea was transferred to the anhydrides of glucose and of cellobiose, either of which was then assumed to be the possible "building unit" of cellulose. Thus, *Karrer*[8], in 1921, expressed the strong belief that the anhydroglucoses in starch as well as in cellulose are held together by means of "secondary bonds" and that here polymerization does not cause structural changes as would be the opening of oxygen linkings.

For Karrer the building unit of cellulose was the anhydro-cellobiose, and the exponent of polymerization (degree of polymerization), deducted from the heat of combustion of starch and from a general analogy with starch, was thought to be not greater than three, probably two.

Thus, Karrer considered the cellulose molecule to be a dimeric cellobiose anhydride $(C_{12}H_{20}O_{10})_2$. This formula denies the possibility that hydrolysis renders other oligosaccharides but the one, namely cellobiose, a postulation which was in agreement with Karrer's strict refusal of any chain structure.

That the cellulose molecule should be represented by only two cellobiose anhydrides held together by secondary valencies, instead of hundreds or more of such molecules linked together by means of normal valencies, certainly was a daring step, even in the memorable year 1921, when the first results of X-ray investigation were thought not to be in disagreement with the conception of the small units. *Herzog* and *Jancke*[9] had found that the group $(C_6H_{10}O_5)_4$ repeated itself regularly within the space lattice of the cellulose fiber gained from monochromatic X-radiation, a result which was welcomed by Karrer as being in good agreement with his conception. This shows that at that time X-ray possibilities were much overestimated, the elementary unit of the crystal was confounded with the molecule.

Still further than Karrer went Hess when he claimed that the cellulose molecule is monomeric, i.e., is represented by a single glucose anhydride. As such it was and apparently still is assumed by *Hess*[10] to exist in solution, for instance in cuprammonium or, as was claimed a few years later (1926 and the following years), in form of derivatives as acetyl- and methyl-cellulose in their respective solutions. In its solid form, also in that brought about by regeneration from solution, cellulose then represents an association of monomeric glucose anhydrides, and such an association is held together by secondary valencies. Association was thus substituted for polymerization, which means that the latter's principle as the outcome of a chemical process was abandoned.

In Karrer's cellulose structure representing a dimeric anhydrocellobiose the two anhydrocellobiose units are thought to hold together by secondary valencies, and a great number of dimeric molecules are thought to be united by means of "crystal" valencies to build up the cellulose itself. Depolymerization then would force to open firstly the crystal valencies, and single cellulose molecules would result. The next step would be the breaking up of the secondary bonds, and single anhydrocellobiose molecules would result. On hydrolysis these would render cellobiose and further, glucose. Although the opinion was expressed that with cellulose depolymerization can scarcely occur without hydrolysis at the same time[11], it was *Hess*[12] who claimed to have obtained (by means of acetyl chloride) an anhydrobiose as a monomeric individual—a claim which seemed to receive much support by the isolation of another anhydrobiose which was claimed by *Bergmann* and *Knehe*[13] (1925).

In case the cellulose molecule were thought to be represented by an anhydroglucose, depolymerization would go the same way, thus rendering single glucose anhydrides before hydrolysis would convert the latter into glucose. Hess thus considered a solution of cellulose to contain single glucose anhydrides. They are, however, inclined to associate together to larger complexes, which makes it difficult to determine their molecular weight by usual means.

Without accepting the idea of the small units, the concept of depolymerization was further developed[14] when it was tried to explain what happens to the cellulose during a number of technical processes, be it the process of mercerization as applied in the textile industry and, further, the conversion of cellulose into solutions and the regeneration to artificial silk, to films, and the like, or be it the isolation of cellulose from plant material by the various processes of pulping, bleaching, etc. It was as-

sumed that with all these processes larger complexes of molecules are broken up into smaller ones without change in the chemical structure of the molecule. The degree of depolymerization, depending upon the intensity of the means applied, expresses itself in a change of various physical properties.

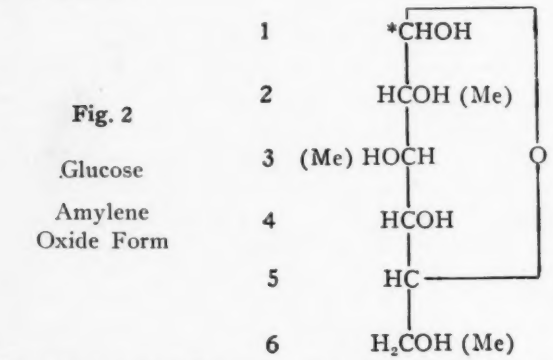


Fig. 2

Glucose

Amylene Oxide Form

It appeared plausible to assume that by the various processes supermolecular forces which hold the molecules together in the various forms in which cellulose appears (as fiber and as amorphous cellulose), are loosened and broken down, which results in smaller complexes, and that this happens long before a change in the chemical structure of the cellulose becomes identifiable. How this conception may well be brought into harmony with the revival of the chain structure will be seen later.

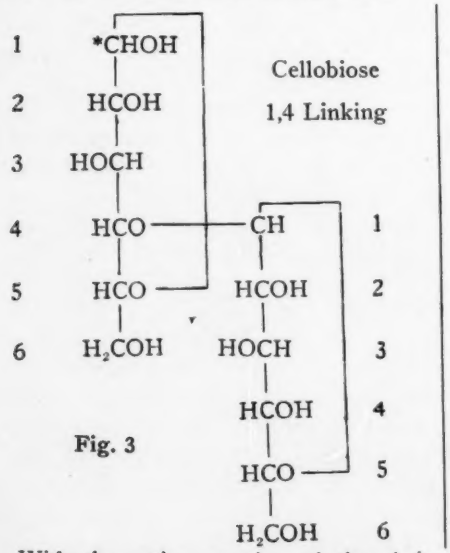


Fig. 3

Cellobiose

1,4 Linking

With the entire negation of the chain structure, as evident in Hess' monomeric anhydroglucose hypothesis, there is only one explanation left for the occurrence of cellobiose, namely, that it synthesizes itself during acetolysis by a process of "reversion," an explanation which, of course, is highly improbable.

In the meantime, the methylation of cellulose which *Denham*[15] had begun in 1913, was made the subject of renewed studies. Hydrolysis rendered trimethylglucose whose structure as that of a 2, 3, 6- methylated monose was recognized by Denham, confirmed by *Haworth* and *Leitch*[16], and later by *Irwine* and *Hirst*[17]. This recognition, together with the fact that Irwine and Hirst could obtain a yield of about 80 per cent of 2, 3, 6- trimethylglucose was of importance, since it was capable of showing that in cellulose the hydroxyls of 1 and 4 of one glucose unit must be occupied. With cellobiose being known as one of the intermediate products of hydrolysis, there was no doubt that one, probably the hydroxyl in position No. 4, was in glucosidic linking with the other glucose unit of the cellobiose, and it was only natural to assume that in this second unit (the non-reducing component) the hydroxyl in position No. 4 was in the same type of linking with a third glucose molecule (*see Fig.* 2, 3 *and* 4). Hence cellulose must consist of at least 3 glucose anhydrides and acetolysis should theoretically render two-thirds of cellobiose and one-third of glucose. But whether the linking is a 1,4 or a 1,5 could not yet be told with certainty. It may be inserted here that already in Tollens' chain formula it is in positions 2, 3 and 6 that the hydroxyles are available for methylation (or acetylation, etc.) whilst the positions 1, 4 and 5 are involved in linking the individual glucose units. Thus, Tollens' old and much contested formula would well explain the formation of 2, 3, 6-trimethylglucose from trimethylcellulose on hydrolysis. (Fig. 5).

Although thus cellulose chemistry made good progress in the direction of chemical structure, the influence of the idea of the small units had reached even the rather conservative English chemists, for *Irvine* and *Hirst*[18] had suggested the cellulose molecule to be built up of 3 glucose anhydrides united to a ring formed triosesaccharide, in which the glucose anhydrides, still assumed to be of the buthylene oxidic structure, were connected up by a 1,5 linking.

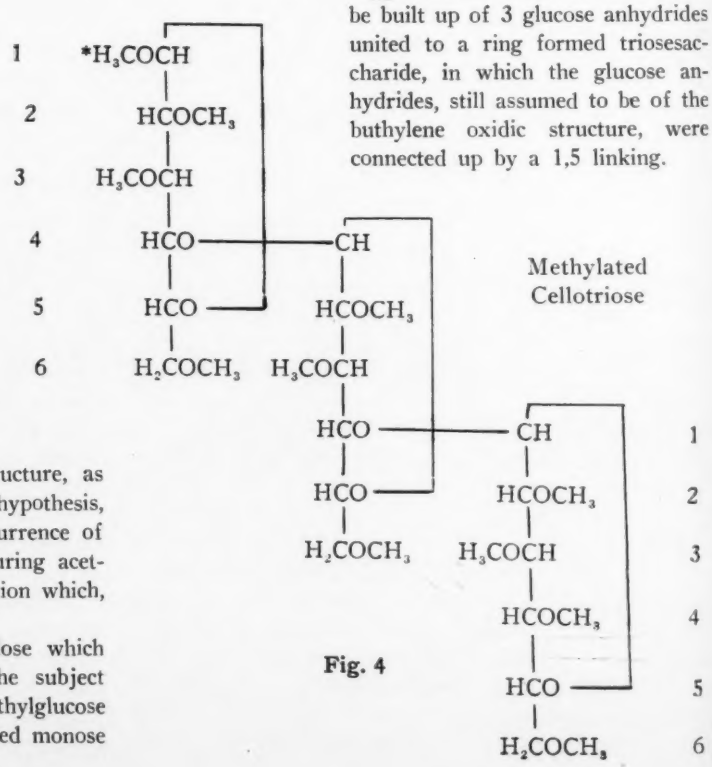


Fig. 4

Methylated Cellotriose

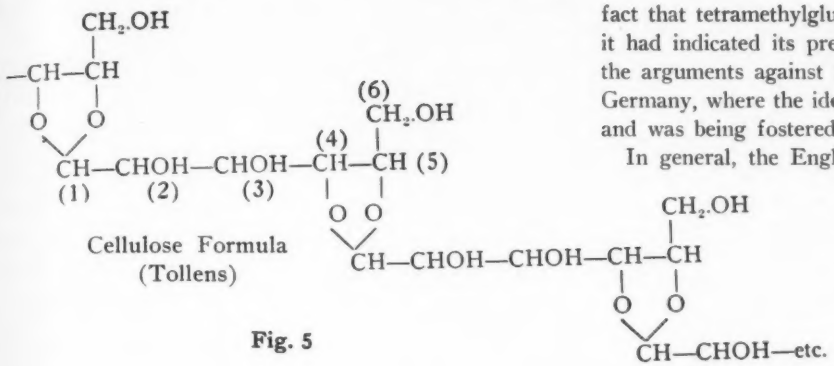


Fig. 5

Ring structure was assumed also to follow from the impossibility to detect any tetramethylglucose. An open chain naturally should have a beginning and an end, and the terminating groups would be each linked only to one neighboring glucose. This being the case, it follows that in the 2 terminating groups there should be, in each of them one more reactive group open to methylation (*see Fig.* 4). In the reducing unit it is the reducing group which is open to methylation and in the other, the non-reducing unit, it is the hydroxyl in No. 4 position which will be methylated. On hydrolysis only the methoxyl of the reducing group is split off, and the latter is regenerated, whereas the methoxyl group in the other terminating unit remains. Thus, hydrolysis would have to yield a certain percentage of tetramethylglucose. Since the latter could not be found, ring structure was assumed.

Here we have to mention *Hibbert's* suggestion made about one year earlier[18]. His formulation of the glucose anhydride was the first attempt to account for the exclusive formation of 2, 3, 6 trimethylglucose (on hydrolysis of methycellulose), and it is interesting that his formulation is identical with that of the β-glucosan which is formed on distillation of glucose under vacuum (see later). Hibbert left the number of glucose anhydrides open for discussion.

It must be emphasized that Denham, when he made his second investigation, already in 1917, had found a trace of a crystalline substance which "resembled tetramethylglucose." Although he did not pay much attention to it, he thought it possible that tetramethylglucose may be "a normal product of hydrolysis of methylated cellulose and that cellulose itself may chiefly be represented by an open chain of condensated glucose radicals." It is unfortunate that Denham made no further effort in this respect, for he, no doubt, would have saved the cellulose chemists a great deal of worry.

On the other hand, the search for the tetramethylglucose was never given up. As already mentioned, Irvine, together with Hirst, looked for it in vain and so did others after them, for instance, *Freudenberg* and *Braun*[19] and it was not until 1932 that *Haworth* and *Machemer*[20] discovered it definitely.

At the time of which I am speaking, 1920-1923, the fact that tetramethylglucose could not be found, although it had indicated its presence dimly before, was added to the arguments against the chain structure, particularly in Germany, where the idea of the small units had been born and was being fostered more than anywhere else.

In general, the English chemists did not bother much about the new conception, the less so since *Sponsler*[21] and other X-ray investigators had made it clear that the elementary cell of the crystal lattice and the molecule of cellulose are two different things and were steadily recognizing the necessity of bringing their evidence into harmony with the results of research on the chemical structure which, at that time as we have seen, strongly pointed in the direction of a chain.

It was the time when the constitution of cellobiose and isomeric disaccharides such as maltose, etc., was in the focus of interest. In 1926, *Haworth, Charleton* and *Peat*[22] gave proof to the constitution of cellobiose as a 1-glucosido-4 glucose (*see Fig.* 3), and in the same year Zemplén[23] arrived at the same structure by way of cyanhydrin synthesis.

About one year earlier, *Haworth*[24] had suggested a revision of the structural formula of glucose. Based upon the fact that normal tetra-methylglucose gives a β-lactone on oxidation, all sugars existing in nature must be given the amylene-oxidic form (five-atom ring). Later, these types of sugars were given the hexagon ring modification (six-atom ring) as shown in Fig. 6.

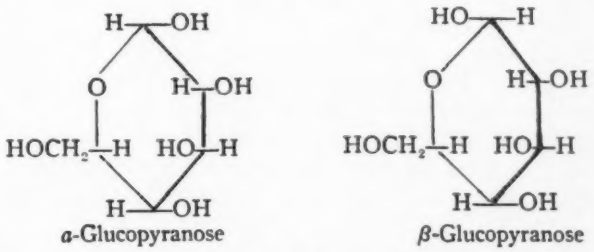
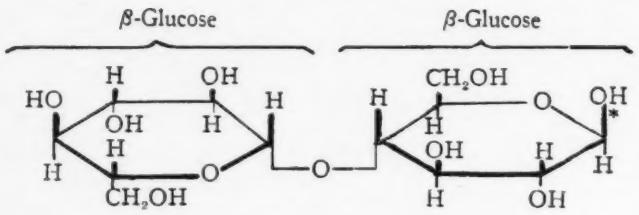


Fig. 6

Due to the similarity with pyrane, which is found to be the nucleus in many natural products, the saccharides were termed "pyranoses," involving a conception which has been accepted since. As will be shown later, Sponsler and Dore and later Meyer and Marck came to the conclusion that they could very well interpret the X-ray diffractions in the light of this new structural formula of glucose.

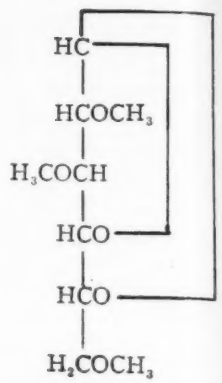
Using Haworth's pyranose structure of glucose, cellobiose is expressed as in Fig. 7.

The importance of the clarification of the cellobiose structure as well as of the new formulation may be expressed in Haworth's own words: "The elucidation of the ring structure of sugars gave a new impetus to consti-



Cellobiose (β-form)

Fig. 7



2,3,6—Trimethyl-glucose anhydride

Fig. 8

tutional study, and the allocation of the hexagon formula to glucose provided new interpretations of the experimental evidence bearing on the constitution of the polysaccharides."

It was a lucky coincidence that at about the same time (1926) the search for oligosaccharides higher than cellobiose amongst the products of cellulose hydrolysis resulted in the isolation of a crystallized trisaccharide by *Ost*[25] and about the same time by *Irvine* and *Robertson*[26]. Already in 1923 *Bertrand* and *Bénoit*[27] had described a triose amongst the products of acetolysis and, as we know today, still earlier, namely in 1913, *Willstaetter* and *Zechmeister*[28] had isolated a triose and also a tetrose from the mixture which resulted from the hydrolysis of wood by means of strong hydrochloric acid[29]. Irvine and Robertson obtained the trisaccharide by interrupting the acetolysis immediately before the formation of the octacetate of cellobiose occurs, which in the old terms of cellulose chemistry means, before the last step of the formation of cello-dextrines was reached.

Probably this is a somewhat later stage than that from which Hess and, about the same time, Bergmann and Knehe believed to have isolated a bioseanhydride. However, these bioseanhydrides were later found[30] to consist of a mixture of longer and shorter links of the chain, a mixture, which, as Freudenberg has pointed out, may well crystallize uniformly[31]. Such may also be the case with Hess' crystallized acetyl-, methyl-, and nitrocellulose preparations[31].

The formation of anhydrides when cellulose is exposed to certain reactions, is of course quite possible. But these anhydrides no doubt owe their formation to a secondary reaction. That is to say, whether it be by means of dry distillation as it was applied by *Pictet* and *Sarasin*[32] and which resulted in the discovery of the laevoglucosan (β-glucosan) or by other means, we have to assume that hydrolysis to mono- or disaccharides is the primary reaction and that the loss of water follows.

These also are the phases of reaction which took place when *Freudenberg* (1927) isolated 2, 3, 6, trimethylglucose-anhydride[33]. (*See Fig.* 8.)

On treatment with hydrogen chloride in ether trimethylcellulose renders 1-chloro-2, 3, 6-methylglucose which on removal of the chlorine by means of sodium metal, is converted into the 2, 3, 6 trimethylglucose anhydride. This anhydride behaves very differently from the trimethyl-cellulose; it has a melting point, a molecular weight in accordance with its monomeric formula, and it lacks any ability to associate itself to higher aggregates. Hence, there is no reason to ascribe to the glucose anhydride and other *lower* anhydrides, obtained from cellulose, such supermolecular forces as would effect association of single glucose anhydrides to the cellulose itself.

On the other hand, polymerization of anhydro-sugars to complex compounds, such as for instance was observed by *Pictet* and *Ross*[34] on β-glucosan when heated to high temperature in the presence of zinc-chloride, or the reassociation of the triacetate of a glucose anhydride from its solution in water to a cellulose-like substance, called "cellosan," as reported by *Pringsheim*[35]—such observations no doubt suggested the possibility of molecular aggregation by forces other than that which we utilize for synthesizing di-, tri- and higher saccharides. But in the case of Pictet's β-glucosan no other clear relation of the complex bodies to cellulose has yet been established and in the case of the association of Pringsheim's glucose anhydride to cellosan it is doubtful whether the primary process, namely the transformation of the cellulose into a glucose anhydride on heating in naphthalene, has actually gone so far, since the determination of the molecular weight, on which the conclusion was based, is unreliable.

In the light of the conception of physical depolymerization, as explained before, we may assure that such processes first break down the bonds which hold the chain molecules together in their higher aggregates as are the micellae. If the processes are sufficiently severe depolymerization will not cease at these bonds but will proceed further and attack the chains splitting them into shorter molecules. The products of such cleavages, however, are still so large that the characteristics of high-molecular substances are still evident.

An attempt to illustrate this principle was made by *Heuser* and *Hiemer* (1925) on various more or less degraded cellulose preparations from cotton cellulose, regenerated cellulose, hydrocellulose, down to cello-dextrines. They were methylated to an extent at which they became soluble in water and the aqueous solutions served for the determination of the degree of depolymerization in various ways. It was found that the latter clearly depends upon

(Continued on page 80)

PROCEEDINGS OF THE
American Association of Textile Chemists and Colorists
FEBRUARY 10, 1936

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WILLIAM H. CADY, *President*
U. S. Finishing Co., Providence, R. I.

A. R. THOMPSON, Jr.—ELMER C. BERTOLET, *Vice-Presidents*

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Lowell Textile Institute, Lowell, Mass.

LOUIS A. OLNEY
President Emeritus and Chairman of the Research Committee
Lowell Textile Institute, Lowell, Mass.

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Midwest Section
Arthur T. Brainerd, Chairman, Ciba Co., 325 W. Huron St., Chicago, Ill.
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South-Eastern Section
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W. F. Crayton, Secretary, Dimon Apt. Columbus, Ga.

Lowell Textile Institute Student Section
Herbert A. Wormwood, Chairman; Henry S. Anthony, Secretary

North Carolina State College Student Section
A. E. Shumate, Chairman; J. W. Furr, Secretary

Members should notify the **Secretary** immediately of all address changes

Fifteenth Annual Banquet*

The meeting convened at eight-fifteen o'clock, Mr. A. R. Thompson, Jr., acting as Toastmaster.

Toastmaster Thompson: Ladies and Gentlemen: I would dislike very, very much to apply the remedy that a certain mother found it necessary to apply to her unruly son when he misbehaved and talked out of turn. She had to take him to the wood shed with one hand and in the other hand she had the hair brush. She laid him over her knee and proceeded to thrash him. It seemed to the little boy that she would never let up. It reminds me of the way some are doing here tonight—it seems you will never let up. But when finally she did let up, the little boy retired to the room to which he was assigned, where he went to the mirror and looked at his posterior and said, "Umph!, just as I thought —split wide open." (Laughter)

This meeting is not going to last very long tonight. We will guarantee that you will be out of this room not later than nine o'clock, which is exactly thirty-five minutes from now. Then the banquet hall will be cleared, and dancing will start at ten o'clock sharply. At eleven-thirty, contrary to the program, which you will notice on your tables, they will have the style and floor shows combined. The schedule originally intended that the floor show would be at eleven-thirty and the style show at twelve o'clock. Please be on time for the style and floor shows.

This job of "toastmastering" is right much of a job. If you have not tried it, I should like to have you try it sometime. It is made particularly difficult by a lot of voices in the back part of the room, and I think probably that is why Harold Schroeder wished the job on me. He told me of one occasion in Chattanooga when he was serv-

*Read House, Chattanooga, Tenn., December 7, 1935.

TOASTMASTER

A. R. Thompson, Jr.

ing as toastmaster at a Democratic rally, to which he had invited the Democrats with only one exception. A very, notorious Republican, none other than Bob Wheeler, was invited to this Democratic rally. Harold would introduce one speaker after another, and each speaker would in turn laud the administration in Washington to the skies. There was little that could be said in addition to the remarks that were made by the various speakers.

Bob was not called upon until the very last, and when he got on his feet, he said: "Mr. Chairman and Gentlemen of the Democratic Party: There is little that I could add to the remarks that have been made concerning the administration in Washington, but what you have said reminds me of a poem entitled 'Mary and the lamb.'" With that he sat down.

Well, they could not get any point to that and so the whispers went from table to table, "What has Bob in mind about Mary and the lamb?" Finally it got back to Harold Schroeder, who was "toastmastering" on the occasion. He was sort of expecting a little trouble and he rather reluctantly called upon Uncle Bob to tell a little bit more about Mary and the lamb. So Bob got up, took a little piece of paper out of his pocket, and read the little poem that reminded him of what had been said regarding the administration:

Mary sheared her little lamb,
And put the wool in bags,
The AAA got all the wool,
And Mary got the tags.

(Applause and laughter.)

That seemed to remind Bob of what the textile industry got at the hands of Washington.

Exactly five years ago tonight, we held our convention in the city of Chattanooga in this very room on the 7th of December. On that occasion a certain gentlemen welcomed us to the city of Chattanooga. He made that welcome so strong that it has been remembered during the past five years, and that is why we see so many from distant places here tonight.

This particular party, I understand, has been the head of your city for nine years. I am told that he has been in public life in Chattanooga for fully thirty years. Thus you will see that your first speaker is amply qualified to welcome you to Chattanooga. You probably wonder who that is, well, the first speaker of the evening will be—(The orchestra played a few bars of "The Old Gray Mare.") Gentlemen, you are wrong. The Mayor is what he used to be and he is only turning gray. It gives me a great deal of pleasure to present to you tonight his Honor the Mayor of Chattanooga, The Honorable E. D. Bass. (Applause).

Remarks by

Hon. E. D. BASS

Honorable E. D. Bass: Mr. Toastmaster, Ladies and Gentlemen: I do remember with much pleasure that it was my privilege to welcome this convention to Chattanooga some five years ago. A lot of water has gone over the dam since that time.

I do not know what has happened to you, but since that time I have been through two campaigns, and I congratulate the people of Chattanooga that you are back here tonight, and I congratulate myself that I am back here with you. (Laughter)

Those of you who were here five years ago I am sure will remember that the best part of my speech was its brevity, and I assure you that I shall be very brief in my remarks tonight.

When I was a boy, we had public hangings in Tennessee, and I remember one time when there was a fellow to be hung here. They took him over to one side of town to the scaffold. The hanging was fixed for eleven o'clock. It seems that the sheriff got there just a few minutes early and he turned to this prisoner, who was a very stubborn sort of fellow, and said, "John, you have ten minutes yet to live. Would you like to say anything to this audience gathered here?"

John said, "No."

Just then a very ambitious young man who was a candidate for the Legislature at that time, arrived and he said, "Sheriff, I should like to use the gentleman's time." (Laughter)

Then the prisoner said, "Wait a minute, sheriff. I came here to be hung, not to be tortured. Go on with the hanging." (Laughter)

I am fully aware that you did not come here to hear me make a speech. I cannot talk to you about your profession. I know very little about it, except that I know it has a very important relationship to many of our industrial assets in Chattanooga. We appreciate your visit here, and we appreciate the fact that you remembered us well enough to came back here to be with us again.

I have learned this since I have been in public life—the

less I say, the better off I usually am, and I do not try to talk about things usually that I know nothing about.

Some three years ago the Governor of Tennessee did me the honor of offering me the State Highway Commissionership for this state. Naturally, I was very much complimented. I told him I would think it over and let him know the next day.

On the way home I drove back from Nashville. Naturally, having that on my mind, I observed the construction of the highways in that part of the state. My attention was called to a sign, by the Highway Department, directing us to look out for soft shoulders. A little further along, I saw another sign reading, "Dangerous Curves." A little farther along, I saw another sign reading, "Men at Work." Half a mile further, another said, "Look out for Children."

I decided that was not the job for me and I refused it. Those suggestive signs didn't appeal to me. (Laughter)

As I said a while ago, I do not know much about your profession. I think perhaps I do know this much, that it might be likened to the little boy who had a stepfather. He said to his mother, "Mother, is your husband my stepfather?"

She said, "Yes, my lad."

"I am your lad?"

"Yes," his mother answered.

"Then I must be the step ladder." (Laughter)

I do know that your profession is probably the step ladder for the success of many of our industries here. We are mighty glad to have you in Chattanooga. We trust you will find time, apart from the duties of the occasion of your visit, to learn something of our people and our city, and of our wonderful civic surroundings. We have a cosmopolitan citizenship in Chattanooga.

A great many years ago sons of the North and sons of the South gathered here and joined hands and resolved to make this a great industrial city. How well we have succeeded you perhaps know, and our success in the past has been indicative of our ambition for the future.

We are mighty glad to have you with us, and we trust the memories of your visit and the recollections will bring you back to us again. I assure you that for you the latch string will always be on the outside.

Mr. Toastmaster, I have another engagement; I shall have to leave you. I wish you a very pleasant evening and Good Night!

. . . The audience arose and applauded. . . .

Delegate: In behalf of the AATCC and in behalf of our Mayor, I bid you all welcome to Atlanta next year for your meeting. (Applause)

Toastmaster Thompson: We appreciate the invitation to Atlanta for next year. Unfortunately, we are unable to tell you that we can accept, because we have accepted an invitation to a city in the East for next year, but we do hope that the Section represented by the vicinity of Atlanta will be in a position to entertain the National Association in the not too far distant future.

We certainly appreciate the invitation and we shall be glad to accept it, I am sure, in later years.

Delegate: I thank you.

Toastmaster Thompson: This morning at our business meeting, we elected a new President. I wish that he could have been here tonight, in fact, for this entire meeting, and heard the welcome that was extended to his organization. However, unfortunately he is unable to be present tonight, and he sends his regrets.

The man I refer to is none other than—(The orchestra played a few bars of "K-K-Katy."). The members of the orchestra are wrong again. It isn't "Katy"; it's "Cady"—William H. Cady. He is the new President of the American Association of Textile Chemists and Colorists. The strange part of it all is how a Republican could be elected President of this Association in Chattanooga. (Applause)

It reminds me somewhat of the little Jewish lad who left Germany and went to Rome, where he went to the Vatican to apply for a job. He was told by the authorities at the Vatican that only a Catholic could be considered for a job in the Vatican, and that, therefore, he had better move on. But this little Jewish lad was not to be outdone, and so he returned in about two months. He said, "Folks, it is all right now; I am 'confoimed'; I am a Catholic and I want my job." He seemed so persistent, and his enterprise seemed to appeal to the authorities, that he was told that they would take him on. The job that was given to him was to knock on the Pope's door every morning and announce the hour and the condition of the weather. The first morning he knocked on the Pope's door at six o'clock and said, "Your Holiness, it is 'seex' o'clock, and the weather is fine."

The Pope said, "Thank you. Well do I know it and well does God know it."

The next morning the Jewish boy, as was his practice the day before, returned and knocked on the Pope's door with the same announcement, without a single change, and the Pope replied in the same way, saying, "Well do I know it and well does God know it."

The third morning the little Jewish lad knocked on the Pope's door and said, "Your Holiness, it is 'seex' o'clock and the weather is all right."

And the Pope said again, "Thank you. Well does God know it and well do I know it."

But as the little boy was on his way out, he said, "And a couple of wise guys are you, because it is nine o'clock and raining like hell." (Applause)

It is getting a little bit near closing time now, but mind you, folks, do not get nervous. We are going to get out at nine o'clock.

The two main speakers on tonight's program are—(The

orchestra played a few bars of "Only a Rose."). No, it is "Olney and Rose." (Applause). We shall hear from them one at a time. (Laughter)

No meeting of textile chemists in this country would be complete unless those present heard a few words from the founder of our Association, from the first President of our Association, from the President Emeritus of our Association, from the Chairman of the Research Committee, who has spent so many hours and so much time on the research programs that are being carried out in different parts of the country.

It gives me a great deal of pleasure, a most unusual pleasure, to present to you a most distinguished educator —Dr. L. A. Olney, President Emeritus of the AATCC.

. . . The audience arose and applauded. . . .

Remarks by

Dr. LOUIS A. OLNEY

Dr. Louis A. Olney: Mr. Toastmaster, until I saw my name on the list of speakers, I thought that I had been successful this time in keeping off of that list and that I could remain in the background and enjoy the evening. However, it appears that I was about as successful as a man of whom I recently heard although the incident itself dates back to the horse and buggy days. He lived in town and his business took him frequently into the outlying districts, so he decided to buy a horse. He took one up on the recommendations of the salesman but the first time he went out on one of his business trips he met with a rather difficult and different experience from what he had anticipated.

It seems that the only possible way of getting out of this particular town was by means of one of four bridges. He drove up to the first bridge and spent some fifteen minutes in attempting to get the horse onto the bridge. He finally gave that up and went to the second. There he was a little more successful; he got the horse on to the bridge for a few feet, but he finally backed off. He then tried the third bridge and the fourth bridge without any greater success. Disgusted and defeated, he turned back and put the horse up in the stable. The next morning there appeared an ad in the local paper which read something like this: "Horse for Sale." It then went on to describe the very fine points of the horse which had been vouched for by the dealer and ended up by saying, "Reason for selling: Owner Must Get Out of Town." (Laughter.)

This business of after dinner speaking, as such, is not a pleasurable one, I assure you. We sometimes think that when the audience laughs and gives us a good hand when we are through talking, we have been quite successful, but what an awakening a few days later when we see the stenographic report and find out how dumb the speech really was. We are then quite humiliated.

How often we mean one thing and say something entirely different. I think that is aptly illustrated by the experience of the young man who recently graduated from a technical school and had the good fortune to secure a job with a prominent and progressive corporation. The president told him that he might spend the first month in just going around through the plant and observing the way things were handled. He did this and at the end of the month he thought he would have a conference with the president. So he arranged for it and began to tell the president just what he had seen here and there that he thought could be improved and how this and that was done wrong, and so on.

The president listened very quietly for a while but soon began to be uneasy. Finally he blurted out, "Who do you think you are? Do you think you are the president of the company?"

"Why, no, of course I don't think I am the president of the company," was the answer.

"Well then, stop talking like a fool." (Laughter)

My interests have been largely, as you know, for the last few years, occupied with the work of the Research Committee. From a very humble beginning, we have grown to a point where we now have six full-time research associates and four assistant associates engaged on five different research projects, which are being carried on in different parts of the country. I scarcely need mention the one which has recently been begun right here in your own city, at the University of Chattanooga, a research with respect to mercerization. Mr. Edelstein is the associate who is directly in charge of the experimental work. I wish to take this opportunity to thank the authorities of the University. They have not only given us half of a whole floor of their laboratory building for this special work, but in addition have offered to furnish apparatus up to $500 for the carrying on of our work.

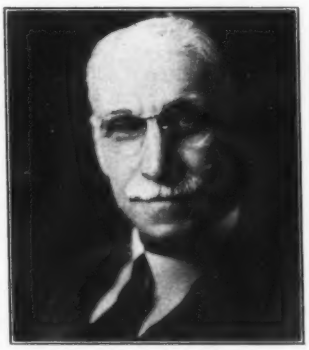
We have at the present time nearly thirty sub-committees, working under the direction of the Research Committee which are actively engaged upon different subjects and undertakings of interest and value to the processing branches of the textile industry.

We have recently taken up the matter of a committee on chemistry of cotton and we believe that the projects which this committee will inaugurate will be of particular interest to the Southern members. Our budget for the work of the Research Committee during the next year will be somewhere around $20,000. More than half of that amount is being contributed by the Textile Foundation. The Chemical Foundation is also furnishing funds for another associate; the Eavenson and Levering Company in Philadelphia are providing for another. Quite recently the Throwsters Institute has appropriated $2,500 and we have secured an equal amount from the Textile Foundation to match this sum. We expect that during the coming year these latter funds will provide for extensive research upon the subject of

rayon, particularly in regard to the creping of fabrics.

The support of our work, in addition to the various sources I have mentioned, comes fundamentally from our corporate members. We are hoping that through the research project on mercerization and the proposed researches in regard to cotton, we may do such creditable work that we will interest some of the Southern manufacturers in becoming corporate members of our Association.

PRESIDENT EMERITUS



Dr. Louis A. Olney

I understand that in this neighborhood there are a great many hosiery manufacturers, all of whom have dye houses, and we believe that the members of this Section might do a great deal during the coming year in increasing the membership from that source. I understand that the Piedmont Section has had the largest increase in membership of any Section in the whole Association. (Applause.)

It would be a very fine thing if this Section could duplicate this record during the coming year.

I was allowed only two or three minutes, but I understand that I am to act also for Mr. Cady, so perhaps if I take a minute more, it will be perfectly satisfactory.

A telegram came for Mr. Cady today, and I took upon myself the responsibility of opening it. I am very glad I did. I shall read that telegram:

"William H. Cady, President
"American Association Textile
"Chemists & Colorists
"Read House
"Chattanooga, Tennessee.

"In electing you President, your Association has given you well-earned recognition for your untiring efforts in its behalf. I add my congratulations, pledge my cooperation, and express my best wishes to you for a successful administration. It is to be regretted that I have been unable to attend the meeting and enjoy the hospitality of our host in beautiful Chattanooga.

"ELMER C. BERTOLET."

Elmer C. Bertolet, as most of you know, was elected a Vice-President of the Association this morning. I wish not only to second this sentiment expressed by Professor Bertolet, but also to say that I have had extended experience with Mr. Cady's work in the Association.

He has been one of the most untiring workers with whom I have had the pleasure of associating. His work in connection with the Research Committee and the Year Book Committee and in almost every activity of the Association has been extremely valuable, and while we regret deeply the retiring of Dr. Rose from the presidency, I think we should congratulate ourselves that the Association is to be continued in such good hands during the coming year.

In closing, I wish to say that it has been a great pleasure to have met so many of my old friends in the South, whom I do not meet very frequently. I wish also to express the great pleasure that I have had in visiting Chattanooga again, and in receiving the cordiality and hospitality of all of the members of the South-Central Section. (Applause.)

Toastmaster Thompson: There have been a few stories told here and I am sure that before daylight, a good many more will be told in other places, so I am not going to attempt to tell another. I am merely going to present to you another man whom you know very, very well. He is none other than our retiring President, Dr. Robert E. Rose.

. . . The audience arose and applauded . . .

Remarks by Dr. ROBERT E. ROSE

Dr. Robert E. Rose: Mr. Toastmaster, Ladies and Gentlemen: I am very happy to be able to say that the ladies have carried all before them and have made sure that in the future they will be a very delightful addition to our meetings.

I am not to face the hangman's noose of His Honor the Mayor but the guillotine, so I must get busy and make use of my time.

I have felt during the past days, as we sat in our meetings, that we have been growing more and more serious, taking upon ourselves the responsibility of solving all the problems relating to textiles, and I think it is time we dissipated a little of this foggy atmosphere of seriousness.

I say "we," feeling that those of you who have been not so studious have realized there is no escape from the laws of nature, that reaction occurs in the direction of removing the peaks even of hilarity and substituting depression.

We have become so serious that I feel we are almost ready to assure the public that if they will only give us money enough and time enough we can save them, not by producing perennial youth perhaps, but by providing them

with burial suits that can be guaranteed so perfect that they will be examples of what research can do for the happiness of humanity.

I do not know what right we have to take ourselves so seriously. After all, what are we anyway? "We are we." What does that mean? And if you say, "We are we," I can say, "We are, are we?" and we do not progress.

RETIRING PRESIDENT

Dr. Robert E. Rose

I feel that it might be well in these few moments to get down to fundamentals. If it is permissible to analyze human beings in terms of fundamentals, in as much as in describing characteristics that divide you from me and me from you—well, we avoid fundamentals, don't we?

We come to the question then, "Why is Mr. Thompson Mr. Thompson, Mr. Thompson being much more interesting to Mr. Thompson than any textile question. (Laughter.) Suppose we ask each one of you, "Why are you?" I don't know what your answers would be, but actually we are we because of only two things—heredity and environment.

We cannot do a thing about heredity because we had no choice when it came to choosing our parents, but we can do something about environment and cheer up and realize that things are going very well if we will only believe it.

We really can modify our environment and we can modify ourselves by doing so. After all, I am certain that the soles of the feet of the present generation do not carry the same heavy load of leather that those of our forefathers did, because we do not walk so much. That is only a physical difference, but mentally we feel disinclined to do what walking we do, which is a characteristic difference.

Therefore, I say we ought to go farther in perfecting the methods of science and doing the best we can to be sure that when we are we no more, the wee we's that come after us shall have a little better environment to make certain they will be a little better than we were. (Applause.)

Toastmaster Thompson: Dr. Rose, you have done nobly. Did you folks see what has been condensed into no more than a talk of five minutes? That is a work of art. (Laughter.)

It is one minute of nine and it would be a most serious mistake if we did not recognize the efforts of all the local committees and the ladies that have made this convention the success that it has been and that it promises to be before morning rolls around again.

Therefore, if I may at this time take the risk of going one-tenth of a second over time, I am going to ask that we give a rising vote of thanks to the men that ran this meeting and to no less than the Press, who have put this meeting over in a big way. We solicit the cooperation of the Press at all future meetings. Let us give the ladies and the Committee a good hand.

. . . The audience arose and applauded . . .

Toastmaster Thompson: The Fifteenth Annual Convention adjourns fifteen seconds later than scheduled. (Applause.)

. . . The meeting adjourrned at nine o'clock . . .

MEETING, SOUTH CENTRAL SECTION

THE South Central Section held its regular winter meeting in the Read House, Chattanooga, Tennessee, on Saturday night, January 18, 1936. Officers were elected, as follows:

Walter E. Hadley, Chairman.
Homer Whelchel, Vice Chairman.
John L. Dabbs, Jr., Treasurer.
Murray B. Smith, Secretary.

Special mention was made of the efforts of Mr. Schroeder, the retiring Chairman, in bringing the Annual Meeting of the Association to Chattanooga, and of the competent manner in which the plans were carried out by the various co-workers.

W. I. Pickens of E. I. duPont de Nemours & Co., Inc. presented a paper on "Practical Hosiery Dyeing Processes". A constructive discussion followed.

Thirty-two members and guests attended.

Respectfully submitted,

H. B. KIMBRELL, *Secretary.*

NORTH CAROLINA STATE COLLEGE STUDENT SECTION

J. W. FURR, Secretary of the North Carolina State College Student Section, reports his Section has just had as its guest Hughes L. Siever, southern sales manager of the Borne Scrymser Co., who spoke on the use of the product "Minerol," and explained the processing of cotton with the Bretton Minerol process, which reduces dust.

Proceedings of the American Association of Textile Chemists and Colorists

Peroxide Bleach Baths

*and Some Factors Influencing Their Stability**

By D. J. CAMPBELL†

WHEN a textile material is treated in a hydrogen peroxide bleach bath, we find that the peroxide content of the bath decreases as the operation proceeds. It is customary to refer to the rate at which this decrease takes place as the stability of the bath. If the decrease is gradual, the bath is said to have good stability and if it is rapid, the stability is called poor. The reason that the peroxide disappears from the solution is that there are two things happening simultaneously. One is that the coloring matter in the textile material is being oxidized and the material is being bleached. The other is that oxygen is being liberated from the peroxide and is passing off into the air as molecular oxygen.

Stability is important in all types of peroxide bleaching operations, but in order to keep this discussion within reasonable limits, it will be confined to a consideration of the type of baths employed in the bleaching of cotton. Stability is important to the cotton bleacher because it enables him to exercise some measure of control over the rate of bleaching, to make efficient use of the available active oxygen in the bath and to avoid bleaching under conditions conducive to the modification of the cotton cellulose.

The rate of bleaching depends, to a very great extent, on the rate of decomposition of the bleach bath. It is possible to speed up or retard the rate of bleaching within certain limits simply by varying the stability of the bath. In the bleaching of cotton goods in a kier, where a large amount of cloth is treated in a small volume of bleach bath, it is very desirable to have the bath decompose gradually so that the bleaching will take place slowly and uniformly throughout the entire mass of goods. It can be readily appreciated that in equipment of this type there would be danger of uneven bleaching if the bath were decomposing rapidly. In other types of equipment it is desirable to bleach fairly rapidly so as to secure the maximum production from the bleaching machine. There are several types of operations where this is quite feasible, for example, in rotary and paddle type hosiery machines, slack loop washers, package machines and others. In such machines the bleaching may take place in from one to three hours but in kiers the process should be so adjusted that it takes place in from five to ten hours.

In order to operate the process as efficiently as possible, it is necessary to endeavor to adjust the conditions so that as much as possible of the active oxygen is consumed in oxidizing the coloring matter and as little as possible passes off into the air as molecular oxygen. In general, the conditions which make for good efficiency are to have a bath with good stability and to use the minimum amount of peroxide. In practice it is sometimes necessary to sacrifice some efficiency in order to get maximum production from the equipment.

In any bleaching operation, where the bleaching is accomplished by means of an oxidizing agent, it is necessary to oxidize the coloring matter without oxidizing the cellulose itself. This selective oxidation can be carried out more readily if it is done slowly or, in other words, if a mild treatment is employed. If a strenuous oxidizing treatment is used, there is always danger of modifying the cellulose. Fortunately, peroxide bleaching is a mild treatment and we have considerable leeway as to what we can do.

There are six major factors which influence the stability of a peroxide bleach bath. These are (1) temperature, (2) pH, (3) type of alkalining agent, (4) the cotton, (5) the water, (6) the bleaching vessel. A knowledge of the effect of each of these is valuable to any one doing this type of work, for it will enable him to control the operation so that it meets his own individual requirements.

The rate of decomposition of any alkaline peroxide solution increases very markedly with a rise in temperature. In the case of cotton bleaching, however, we find that it is possible to vary the temperature only within rather narrow limits. In order to obtain satisfactory whites, it is necessary to raise the temperature of the bath to about 180°F. This seems to be the lowest temperature at which the bleaching will proceed to completion for at much lower temperatures, even after excessive periods of treatment, the cotton does not bleach satisfactorily. In most cases, it is desirable to keep the temperature at or near this point. In some special cases where an increase in the rate of bleaching is desired, a rise in temperature is frequently a most convenient method to use, and the bleaching may be done at temperatures as high as 200°F. If the stability of the bath at 180°F is not sufficiently good to meet the conditions, other means will have to be used to control it.

Perhaps the two most important factors that influence

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† R. & H. Chemicals, Dept., E. I. du Pont de Nemours & Co., Inc.

the stability of an alkaline peroxide bath are the pH and the type of alkalining agent. Peroxide solutions are most stable in acid solution but in order to obtain good bleaching, the baths must be made alkaline. The rate of decomposition increases very rapidly as the pH of the solution rises. The following table will give some idea of what happens.

TABLE NO. I
Rate of Decomposition of One Volume Hydrogen Peroxide Solution at 180°F.

pH	*Time Required for 50% Decomposition*
6.8	3.2 hrs.
7.1	2.8 hrs.
7.9	2.2 hrs.
8.9	1.2 hrs.
9.9	25 minutes (approximately)

These figures were derived from a study of solutions containing only hydrogen peroxide and sufficient caustic soda to adjust the pH. Solutions of this type are very susceptible to the presence of small traces of impurities such as metallic ions which cause catalytic decomposition so care was taken to exclude them as far as possible.

If we substitute carbonates, phosphates or borates for the caustic soda, we obtain a series of values very similar to the above and there will be only unimportant variations. None of these materials exert any pronounced stabilizing effect. At a temperature of 180°F sodium pyrophosphate gives results very similar to the ortho phosphate. At lower temperature it exerts a pronounced stabilizing action as compared to sodium hydroxide. It is, therefore, quite extensively used in low temperature work such as in wool bleaching where the bleaching is done at 120°F.

If sodium silicate is substituted for the sodium hydroxide, we obtain solutions which show a marked improvement in stability, as shown by the following table:

TABLE NO. II
Rate of Decomposition of One Volume Hydrogen Peroxide Solution at 180 F. Containing 7.2 Grains per Liter of Sodium Silicate $Na_2O.2.5\ SiO_2$ (pH adjusted with NaOH or H_2SO_4)

pH	*Time for 50% Decomposition*
6.0	3.75 hrs.
7.0	3.5 hrs.
8.0	11.0 hrs.
9.0	5.5 hrs.
9.6	4.0 hrs.
10.5	1.5 hrs.
12	10 minutes (approximate)

Compared to caustic soda, sodium silicate is a stabilizer, for there is an appreciable increase in the time required for decomposition in the pH range 8.0 to 10.5. This is one of the factors that has made sodium silicate the most widely used alkalining agent for peroxide bleach baths.

The amount of silicate used in these baths corresponds to the amount frequently used in commercial bleaching, six pounds to one hundred gallons of bleach bath. If the amount of silicate is reduced by 10% and sufficient soda ash is added so that the resultant solution will have the same pH and total alkali content as the original peroxide silicate bath, the change is immediately reflected in an increased rate of decomposition. If another 10% of the silicate is replaced by soda ash, the rate of decomposition is still further increased. If we substitute tri-sodium phosphate for part of the silicate, the rate of decomposition is not materially increased until over half of the silicate has been replaced. These procedures are frequently used in practice to adjust the rate of bleaching to the desired point.

In all of the baths that have been discussed so far, there has been no bleachable material present, so the oxygen lost by the solution passed off into the atmosphere as molecular oxygen. When grey or boiled out cotton is introduced into these baths, a new set of conditions is set up. The cotton exerts a stabilizing action on the peroxide and in spite of the fact that bleaching is taking place and some of the oxygen is being used in this way, the rate of decomposition is slower. Grey cotton exerts a more pronounced effect than cotton that has been well boiled out and purified prior to bleaching. This stabilizing action is noted in baths made alkaline with any of the commonly used alkalining agents but reaches a maximum in the presence of sodium silicate. The stability of these baths is satisfactory for bleaching over the pH range 8.0 to 11.0 and reaches the maximum at pH 9.8 to 10.2. Therefore, most of our cotton bleaching is done in the range pH 9.8 to 11.0. Below 9.8, the baths are stable enough but the bleaching action is not as good. The combination of grey cotton and sodium silicate is one of the best stabilizers we have for alkaline peroxide baths.

As in any wet processing operation, the water used in preparing the bleach baths is an important item. Plant water may contain both organic and inorganic impurities. The organic impurities may sometimes interfere with good bleaching results by causing stains and other troubles but as a rule they do not influence the stability of the peroxide to any extent. However, the inorganic impurities exert a noticeable effect on the stability of peroxide solutions. The calcium and magnesium compounds present in hard water and the iron which is present in most waters are the most important impurities of this type. Peroxide bleach baths prepared in hard water are more stable than those prepared in softened water. Magnesium is more important than calcium in its stabilizing action and even small traces produce a considerable effect. At first glance, this action might appear to be an advantage but these two metals form many insoluble compounds which may cause trouble in the processes subsequent to bleaching.

The stabilizing effect of hard water is frequently offset by other impurities. This is illustrated by considering the effect of a few examples of plant waters, as shown in

Table III. The figures for the stability were determined by bleaching samples of cotton in peroxide bleach baths alkalined with sodium silicate prepared in the various samples of water and running a similar bath prepared in distilled water as a control. The ratio of the time required for 50% decomposition of the bath prepared in the water under examination to the time required for 50% decomposition of the control was taken as the stability.

TABLE NO. III

Hardness	*Iron*	*Total Solids*	*Stability*
p.p.m. as $CaCO_3$	*p.p.m. as* Fe_2O_3	*p.p.m.*	
12.5	0.5	62	1.1
17.1	0.5	89	1.7
38.6	0.2	97	4.0
70.1	1.2	97	1.6

These figures also shed some light on the effect of iron as well as the hardness. By comparing the first two examples, the improvement in stability seems to be due to the increase in hardness. In the third example, the large improvement in stability seems to be due to the increase in hardness and the lower iron content. In the fourth case, there is a big increase in hardness but the stability is much poorer than in the third example which is probably due to the increased iron content.

Iron must be considered as accelerating the rate of decomposition but it is not nearly as active as some other metallic ions, for example, copper, manganese and cobalt. Fortunately, these three are rarely found in plant waters for there is no effective method of inhibiting their catalytic effect. The effect of iron depends, to a great extent, on the form in which the iron exists in the solution. Sodium silicate plays an important part in inhibiting the effect of iron for it appears to have the property of forming a complex compound with iron which is not ionized and renders it much less active than if it were in some other form, for example, ferric hydroxide.

Silicate also possesses the valuable property of forming films over metallic surfaces, so it serves another useful purpose because much of the cotton bleaching is done in iron kiers and other metal equipment. In kier bleaching the iron surfaces must be covered with a protective coating to prevent corrosion. If this point is observed no difficulty will be encountered. It is customary to coat the surfaces which come in contact with the goods with cement or lime before attempting to bleach in a new kier. The inner surfaces of the circulating pipes and other inaccessible areas are coated by pumping a solution of silicate through them. These coatings are being constantly renewed by the silicate normally used in the bleach bath so that the bleacher should not have to worry about them.

Copper is such an active decomposition catalyst for peroxide that copper tanks cannot be used for bleaching. Nickel is not an active catalyst and containers of this metal can be used. Monel metal apparatus is quite widely used for bleaching equipment, particularly in the hosiery industry. This material increases the rate of decomposition and as a rule, fairly rapid bleaching takes place. Stainless steel of the general type containing 18 per cent chromium and 8 per cent nickel makes an excellent material for the construction of bleaching equipment as it does not materially affect the stability and is not corroded by alkaline peroxide solutions. Lead is used to some extent for equipment but it must be kept coated with silicate, or in baths containing sulfates a coating of lead sulfate is formed. If it is not coated, an accelerated decomposition will result and the goods may be stained. Aluminum is used to some extent and where the baths contain silicate it seems to be giving satisfactory results. Wooden tanks, after they have been used once or twice, do not have any effect on the stability of the peroxide bath and are, therefore, satisfactory.

The peroxide bleaching of cotton is a process that can be controlled and adjusted to work under a wide variety of conditions. In laying out a process to meet any set of conditions, the first thing to be considered is the material itself and the type of process that will be needed to meet the requirements. Many fabrics require a preliminary boil or scour while in other cases the grey goods are simply given a bleaching treatment. Both procedures will produce good whites and the choice will depend upon the use for which the material is intended.

The next thing to be considered is the water. There is seldom any difficulty on this point for most bleachery water supplies are suitable for peroxide bleaching if they are suitable for bleaching at all. The equipment must be inspected and its effect on the stability of the bath considered.

After this preliminary study it should be possible to estimate the amount of peroxide required and select the most desirable combination of alkalining agents so that after a few trial runs, when any necessary adjustments can be made, the process will operate on a regular schedule.

Peroxide bleach baths can be prepared from any of the compounds which have reactions similar to hydrogen peroxide. The most important compounds in this class are hydrogen peroxide itself, sodium peroxide and sodium perborate. For bleaching, hydrogen peroxide and sodium peroxide are the ones most widely used and baths prepared from these reagents will have the same bleaching properties under identical conditions. A solution of sodium perborate will have the same stability as a hydrogen peroxide solution made alkaline with sodium metaborate; perborate solutions are quite extensively used for the oxidation of vat colors. In a general discussion of this sort, no specific suggestion as to the peroxide content of the

baths can be made, as this has to be adjusted in each particular case. Usually it varies from 0.1 to 0.7 volume hydrogen peroxide and it is desirable to adjust it so that the bath will be entirely decomposed at the end of the run so that there will be no waste.

DISCUSSION

Chairman Draves: I am sure we have all enjoyed this paper. Have you any questions to ask Mr. Campbell?

I have one question, Mr. Campbell.

I have dyed skeins on machines with sulfur dyestuffs and obtained a coating of copper sulfate on the Monel lining, and then when I tried to bleach skeins with peroxide I found the peroxide decomposed pretty rapidly.

Mr. Campbell: You will have to use peroxide in there first and clean up the tubs, first. Peroxide will do that with most any sulfide. It is perhaps well to remove the coating and to put one on with silicate if you can afford the time to do it.

MEETING, NORTHERN NEW ENGLAND SECTION

A MEETING of the Northern New England Section was held at the Hotel Manger, Boston, on Friday, January 10, 1936 with 75 present.

Roland Derby, Chairman, presided and introduced L. A. Olney, who gave a brief account of the recent annual meeting at Chattanooga.

B. A. Ryberg, Research Associate of the A. A. T. C. C., presented a paper describing his present work for the Association, entitled "Methods of Analysis of Wool and Cotton Textiles". He demonstrated a proposed method for the separation of cotton from wool which was much appreciated by those present.

Respectfully submitted,

JOHN N. DALTON, *Secretary.*

NOTICE TO MANUFACTURERS AND IMPORTERS OF CHEMICALS FOR THE TEXTILE INDUSTRY

(*Reprinted from January* 13, 1936, *issue*)

IN THE 1933 Year Book of the American Association of Textile Chemists and Colorists there appeared for the first time an Alphabetical List of Textile Chemical Specialties. Now material is being collected for the same list for the 1935 Year Book. Those manufacturers and importers who have not yet participated in this work of the Association are invited to assist us in publishing this year a larger, more complete and more useful list of the chemical specialties which are available in America for the textile trade. If you have products which should be included in the list and if you have not already been canvassed, write at once to the chairman with the information asked for on page 316 of the 1934 Year Book.

CARL Z. DRAVES, *Chairman, Committee for Textile Chemical Specialties, 273 Converse Street, Longmeadow, Mass.*

Proceedings of the American Association of Textile Chemists and Colorists

Synthetic Resins

*Their Application to Textiles**

By D. H. POWERS†

INTRODUCTION

SYNTHETIC resins are a development of the twentieth century. While some of the natural resins are undoubtedly thousands of years old, the synthetic resins have all been produced in the last 25-30 years. The manufacture of these resins has grown from a process for the production of a dark colored product of indefinite composition and behavior, to the production of thousands of synthetic resins of carefully controlled composition and physical characteristics. A resin has been defined as "A solid solution of organic substances." To elaborate further, it is a mixture of solid organic compounds in varying proportions which gives products that are characterized by their lack of definite physical characteristics. In general, they do not have a sharp melting point and are frequently infusible substances. Their more highly polymerized or condensed forms are often insoluble in all organic solvents. They may be film forming and usually flow under the action of heat and pressure. The synthetic resins which are of chief industrial interest and importance are usually organic condensations of resin forming substances which are mixed and sold or applied as molding powders or as permanently fusible resins. These products on drying, heating or exposure to air are quickly converted into much less soluble products. Factors affecting the type of resin formed are: (1) The nature and ratio of reacting chemicals; (2) concentration and type of catalyst used; (3) time and temperature of curing or setting conditions, and (4) presence of diluents or fillers.

RESIN DEVELOPMENT

In reviewing the development of synthetic resins in the present century we find that hundreds of workers and investigators have contributed their part to the improvement and modification of these products and their development from dark brittle solids into clear, crystal glasses. In going over the workers a few names stand out for their pioneering achievements. In 1905-1907 Leo Baekeland actively investigated the phenol formaldehyde resins, their preparation and stabilization. The well known Bakelite molding resins which have found thousands of applications is an industrial tribute to his pioneering efforts and performance. From 1910-1920 Kurt Albert actively investigated the phenol formaldehyde resins. By incorporating rosin and other bodies he was thus able to produce phenol formaldehyde type resins which were soluble in the Oleo-resinous varnishes. With the development of these new synthetic resins with improved solvent solubility he was able for the first time to compete with a natural resin such as Copal and Damar and the synthetics began to directly compete with the natural resins. In 1920 Hans John worked and developed the urea formaldehyde type of resins and starting from these intermediates, he developed colorless, light stable resins which have been of particular importance where crystal, clear or delicately tinted resins are required. As early as 1915 Callahan and much later Kienle worked on the glycerin phthalic anhydride condensations and by combining these products with the various drying oils produced a tremendous range of synthetic resins now classed under the heading of alkyls.

These resins can be produced in almost any desired state or physical characteristic, ranging from a hard, brittle solid to a soft plastic mass. By varying the properties of ingredients, varying solubility and miscibility in solvents is possible and a wide variation in physical characteristics is obtained. In addition to these investigations, there have been a number of other developments of wide interest and importance. Some of the more important ones which deserve mention are the syntheses of the vinyl esters and the acrylates and even products such as chloroprene and cellulose acetate may be considered as types of synthetic resins. The list of synthetic resins is by no means exhausted and their number and types are continually increasing. The new synthetic organic chemicals are being continually investigated and tested to determine conditions under which they may be converted into new types of resins. The development of this field has reached to the stage where a resin of almost any desired characteristic may be compounded and the only limiting factor is price. It is also significant to note that the price of these resins has continually dropped until they are today in common use on thousands of articles sold in the Woolworth stores.

COATED FABRICS

While the resins have been actively developed and studied, and their application widened, little use has been made of them for treating textiles. After all it was reasonable to suppose that a hard, dark colored, brittle solid would have little use and value in a textile fabric which must be soft, adsorbent and flexible. There has been a limited use of the synthetic resins to modify and improve the oil or lacquer coatings. The incorporation of synthetic resins in the oleo-resinous varnishes as well as their uses as plasticizers for nitrocellulose lacquers made possible more flexible coatings with better durability and ageing characteristics. However, coated fabrics of this

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† Röhm & Haas Company.

type cannot be truly considered modified fabrics—but are really sheets of paint or varnish with a textile backing. They are primarily a waterproof sheeting and secondarily a textile fabric. These coated fabrics have lost all of their ability to drape, their adsorbency, softness and the characteristic hand of a textile. The majority of the coated fabrics are spread with nitrocellulose dopes, linseed oil type varnishes or heavily compounded rubber mixes. These coated fabrics have developed their own markets as a covering material, for bridge table covers, as an oil cloth or auto top covering material.

RESIN MODIFIED FABRICS

There appears to be a tremendous field for resin treated fabrics which are essentially fabrics after the resin has been applied. Until recent years there has been no development in this field. The problem has been a difficult one and only years of patient research have made possible a few developments which show promise of producing resin modified fabrics which are resistant to crushing, flexing and creasing. The resin treated fabrics are still similar to textiles but possess specific characteristics which they normally do not show.

RHONITE AND RHOPLEX

The slow development of resin modified fabrics is explained by the complexity of the problem. The same resin with only slight modifications gives entirely different results and effects depending on how it is applied. A surface coating tends to destroy the fabric effect and penetration into the fiber gives the most striking results. On the other hand, resins which show the most promise in sheet form may have an entirely different effect when impregnated into the fiber. We have investigated a new synthetic resin, RHoplex, sheets of which show unusual properties of flexibility, elasticity, tensile strength and freedom from color. However, when impregnated into a fabric it does not produce finishes which are nearly as crush-resistant as the finishes produced by treatment with the RHonite resins. These RHonite resins, on the other hand, are not at all flexible and form hard, solid, glass-like sheets. It has become increasingly evident that the successful development of resin modified fabrics is dependent on a close cooperation between the resin manufacturer and the textile finisher. The resin manufacturer will usually choose the resins which have the best film-forming properties and frequently will not be at all suitable for producing a soft adsorbent fabric. The textile finishers working alone produce surface coated fabrics as they may not use conditions best suited for complete resin penetration.

TYPE OF RESIN REQUIRED

In considering the conditions to be met by the resin manufacturer in producing a resin suitable for general textile application the following points are important. The resin should be:

(1) Free from color.

(2) Fast to washing and soaping.

(3) Show no embrittlement or tackiness.

These three factors eliminate a great majority of the resins. Of the thousands that have been synthesized only a few come within these specifications. A resin with even a faint yellow cast is eliminated as the color limits or prevents their application on white goods. It has been found that the washfastness of resins dispersed in a fabric is very much poorer than its washfastness in slab form. On the other hand, resins which are distinctly brittle such as the RHonites, or resins which may be tacky in sheet form such as the RHoplexes, do not show these characteristics when properly incorporated into the fibers and fabrics.

PLANT CONDITIONS REQUIRED

Considering the problems which arise during the mill application of the resins, the following points are pertinent:

(1) Application in aqueous solution.

(2) Ease of fixation.

(3) Stability of solutions.

(4) Adsorbency of finish.

The application of the resin in aqueous solution is important not only from a point of view of eliminating the cost of an organic solvent, but more especially because it insures the application of a resin which will penetrate into the fiber. The use of water as a solvent or dispersant also eliminates the inflammable solvents which are a fire hazard and saves the cost of a solvent recovery unit. If the resins are applied in aqueous solution, a method must be worked out for quickly making them insoluble and wash fast after they have been put on the fiber. A heat curing process is the method generally used for setting the molding resins. A combination of high temperature and pressure with a suitable catalyst is used by the resin manufacturer to convert the resin into a completely insoluble form in a few seconds. But the pressures involved are several tons and the temperatures required are usually from 300-400° F. For the general textile application of such a resin it has been necessary to work out a catalyst which will effect a curing or insolubilizing of the resin at temperatures as low as 230° F. without pressure.

RESIN STABILITY

One of the serious problems in resin manufacture has been the production of stable resin solutions. By their very nature, they are products which can be quickly converted to insoluble masses. It is, consequently, extremely difficult to isolate and protect them so that they may be stored for weeks and months without any change whatsoever. The resin manufacturer feels that he has come a long way when he has produced a soluble resin form which can be stored three months with less than 3 per cent change and will still change to a completely insoluble form when cured for 60 seconds.

ADSORBENCY OF FABRIC

It is important to note that the adsorbency of a fabric

may not be materially affected even when impregnated with a completely insoluble resin. This occurs only when the resin is impregnated into the fiber, the same resin *on* the fiber will give a completely water insoluble finish. The resins to be wash fast, must be insoluble, but they decrease the adsorbency only when they coat the yarns. It is particularly significant to note that the wet strength of rayon yarns may be greatly improved by proper resin impregnation.

THE ANTI-CREASE FINISH

In considering successful resin applications to textiles, the Tootal Broadhurst Lee Co. of England deserves first consideration. For the past ten years they have actively worked on the problem of proper resin impregnation. As a result of this work they have developed the anti-crease process which has found such wide application abroad and is gaining increasing importance in this country. As a result of their work we find on the market crush-resistant linens, spun rayons, and necktie fabrics, three lines which promise to be the most important fabric developments of the year. In addition to these fabrics there is an active development in the treatment of pile fabrics, suitings and linings in this country.

NEWER DEVELOPMENTS

A number of new developments requiring the use of resins in finishing fabrics are being studied. Some of the more important ones which may be mentioned are the wool finishes on cotton, organdie finishes on sheer goods, and non-slip finishes on rayon flat goods. There is a fertile field here for further development and the production of new unique finishes and effects. But this development will require careful and close cooperation between the yarn and fabric processor or finisher and the resin manufacturer. It is a field where initial failure can almost generally be assured and where continued trial and study of fabric construction, yarn twist, resin condensation, curing conditions, fiber adsorbency, solvent and swelling effects and resin penetration may be expected to yield striking and original results. It is entirely within the realm of possibility to expect as a result of such development a true rayon finish on a cotton core, all of the properties of wool imparted to a rayon or cotton fiber, the scroop and finish of silk on a rayon yarn, a non-felting or non-shrinking finish on a worsted or a woolen. We look forward to the time when we can impart to one fiber the characteristics and properties which we have previously thought we confined to some other. We may also expect to modify the dyeing properties of the yarns by the incorporation of nitrogen, sulfur, carboxyl, or phenolic groups.

CONCLUSION

In conclusion, the application of resins to produce modified and improved fabrics is a very recent one. The production of successful finishes has been dependent on a close cooperation between resin manufacturer and fabric and yarn processor. Recent years have seen the development of a wide variety of resin-treated fabrics with new properties and has seen tremendous improvement in the production of resins suitable to textile mill application to insure deep penetration and quick, convenient fixation. With the promising start which has been made there is reason to hope and expect for many developments which will open new fields to wider applications for our textile fabrics.

DISCUSSION

Chairman Draves: I am sure this paper by Dr. Powers has been extremely interesting. This is one of the outstanding research developments of the past few years and although it was first of importance in England, I am sure that it is going to become of increasing importance in the United States.

Mr. McCarty: You mentioned 15 per cent on that pile fabric. Are you talking with regard to the dry synthetic resin, or the liquid?

Dr. Powers: I am talking about the actual resin in that fiber as it stands there, which that particular fabric happens to have.

Mr. McCarty: It happens to have thirty or forty per cent of the commercial form in it.

Dr. Powers: You can vary your concentration. These resins are water soluble and by varying your concentration, you can use a weak solution or a strong solution, depending upon conditions of application.

Mr. McCarty: You have fifteen per cent of actual resin?

Dr. Powers: Yes.

Mr. McCarty: In treating cloth or yarn with synthetic resin, is it practical to dye it afterwards?

Dr. Powers: It is practical, although generally speaking, it is done as the last operation. In other words, in general resin applications of this sort, a variation of a half per cent in the resin content is not perceptible as a variation of the crush-resistance or the firmness of the fabric, but a variation of one-half, one or two per cent in the resin would have a very definite effect upon the dyeing properties. Consequently, in our work, its application is the last step.

Mr. McCarty: I have formaldehyde resins that are more absorbent to moisture than a finished fabric that has not been finished with synthetic resins.

Dr. Powers: I think it is the cellulose that is doing the absorbing. I am sure that the resin itself is not increasing the absorbency. It may be that the fabric is more absorbent due to the washing of the fabric or careful treatment.

Prof. Grimshaw: Has any work been done upon creasing properties?

Dr. Powers: For preventing creasing?

Prof. Grimshaw: Yes.

Dr. Powers: This is an organdie. This is not the anti-crease finish, but contains surface resin. Now here is an

interesting point and the question arises: if you have a fabric resistant to creasing, can you crease it? What about a high grade worsted? It is one of the most resilient fabrics that we have, yet once you put a crease in your trousers it holds very well. Once you put it in there with steam and press it, it holds better than a fabric which is soft. The same is true of these crease-resistant fabrics.

Prof. Grimshaw: Are these resins soluble in solutions of caustic soda, for example, of mercerizing strength?

Dr. Powers: Well, some are. Of course, these particular resins are, but the great majority are not. You never could cure these resins in alkali. All of the resins require conditions to make them set and this particular type of resin will not set in an alkaline solution. It has to be very slightly acid to throw it over from a soluble form to an insoluble form. These resins are not particularly substantive.

Question: Could quicker results be obtained using solvents in place of water solutions?

Dr. Powers: This flexible resin that I have here, which you can stretch, is available in a solvent or in water. Here is 25 per cent in solvent and here is a 25 per cent emulsion in water. One is a milk and the other is a viscous solution. Generally speaking, the solvent does not give good penetration into a cellulose type of fabric.

We have been doing some work on cellulose acetate and there the use of the solvent tends to help penetration. On cotton, as you know, alcohols do not swell the cellulose and you get much better results with a solvent that will swell the fiber and improve the penetration.

Question: Has any work been done on the application of synthetic resins to rayons in the filament form?

Dr. Powers: Do you mean in the form of yarn?

Question: No, in the form of thread.

Dr. Powers: Well, there is some work being done on that, and the success of that work depends on thoroughly squeezing out the thread after impregnation. With this RHonite resin, for example, you take the powder and dissolve it in water, wet your fabric, your yarn or your thread thoroughly in it; then before you dry it, you squeeze out all the surface resin.

You can run piece goods through a hydraulic calender, to give them a thorough wringing and you get that surface resin removed. On yarn, the same thing is possible.

There is no reason why yarn in single strand form could not be treated, provided you can work out an adequate method of squeezing the yarn and getting that excess off.

It can be done very successfully in skein form by wetting-out the skeins and hydro-extracting them to get the surface resin off.

Mr. Hadley: Can you add this resin to thread without causing the threads to stick together?

Dr. Powers: You will find if you untwist the yarn in this fabric they are not stuck together. The resin itself goes into the yarn and you can untwist your fiber into individual threads. In that particular fabric there is probably twelve or fifteen per cent resin, and yet it possesses maximum softness.

Mr. Hadley: What would be the possibility of treating the raw stock? Could it be dyed afterwards without affecting the resin?

Dr. Powers: Yes. The resin would affect the substantivity of some of the colors. The resin tends to make cotton dye like wool. There is still a lot of work to be done to make it dye more like wool, and the treated fiber has more tendency to take up the acid type colors. Raw stock has been treated very conveniently by wetting it out in an aqueous resin solution, whizzing and drying.

Mr. Hadley: Has anything been done with regard to the use of resin for the production of very light shades?

Dr. Powers: That brings up another problem which is very interesting, namely, the use of resins to fix the colors and they are showing increasing promise.

There are some indications that resin does improve the crock fastness of colors. There again, in deference to the dyestuff manufacturer, it is cheaper to use a better color than to try to cover up the whole face of the fabric with resin, just to improve the crock fastness.

Mr. Hadley: Does the resin tend to retard soiling at all? Does it wash up more easily?

Dr. Powers: The fabric definitely soils less because it isn't so fuzzy and gives a cleaner yarn. With a resin of this type, where you tend to lay the threads, of course, it soils less, but there again it is a difference of degree. It does soil but not nearly as much.

Question: How did you get away from lacquer tackiness?

Dr. Powers: This particular resin has no tendency towards tackiness at all. Generally speaking, in applying water solutions of these resins there is no tendency for the fabric to stick at all.

Question: I had in mind that raw stock proposition.

Dr. Powers: If you treat skeins or raw stock, after they are whizzed, dried and the resin fixed, then pull them apart, they are more resilient, more springy, and harder to weave than cotton. They are more like wool, because of the springiness that you impart to the fiber, but we have not run across any tendency for it to stick the fibers together.

Mr. Manning: It might be possible to make a filament out of something other than rayon, for instance, out of a protein base. The protein base would then be probably insoluble. In order to make a synthetic fiber, it might then be possible to inculcate a resin compound into that fiber, and then dye the fiber with an animal dye instead of a cellulose dye. Of course, you would have to impregnate it before you used it in a fabric, because the material would not stain.

Dr. Powers: That is merely one side of the problem. There are two ways of tackling this problem. There is the problem of proteinizing the rayon and dyeing it like wool,

or the possibility of taking cotton and rayon and dyeing it with cotton colors and resin treating it to impart the properties of wool. It was my feeling that perhaps the logical solution of that problem would be to take the rayon or cotton and dye it like cotton, where the methods are well worked out and then resin treat it to give it the physical properties of wool.

ANNUAL MEETING, PIEDMONT SECTION

THE annual meeting of the Piedmont Section was held in Charlotte, N. C. on Saturday, November 9, 1935 with 275 present for the banquet at the Hotel Charlotte. Several excellent entertainment features were presented. (*Ed. Note*—A photograph of the banquet group was published in the January 13, 1936 issue of the AMERICAN DYESTUFF REPORTER.)

The two chief speakers were B. B. Gossett whose subject was "The Textile Industry" and G. W. Hinkle of the Republic Steel Corp. whose subject was "Stainless Steel in the Textile Industry." Mr. Hinkle illustrated his address with a sound moving picture showing the manufacture, fabrication and application of stainless steel.

Officers for the year 1936 were elected as follows: Chester L. Eddy, Renfrew Bleachery, Travelers' Rest, S. C., *Chairman;* T. R. Smith, Wiscassett Mills Co., Albemarle, N. C., *Vice-Chairman;* Raphael E. Rupp, Pacific Mills, Lyman S. C., *Secretary;* Lawton W. Cleveland, Beacon Mfg. Co., Swannanoa, N. C., *Treasurer;* Charles H. Stone, Charles H. Stone, Inc., Charlotte, N. C., *Councilor.*

Paul Haddock, the retiring chairman, was presented with a cup by the Charlotte Chamber of Commerce in recognition of his efforts on behalf of the Piedmont Section.

Respectfully submitted,
THOMAS R. SMITH, *Secretary.*

DELEGATES OF INTER-SOCIETY COLOR COUNCIL

Announcement of Annual Meeting of Inter-Society Color Council

The Inter-Society Color Council will hold its annual meeting on Thursday, February 20th, 1936, at 7:30 P.M. in the Astor Gallery of the Waldorf-Astoria Hotel, New York City.

Following a brief but *important* business meeting the conference will be open to the members of the organizations that are members of or affiliated with the Council.

At this meeting which will be at 8:00 P.M. the Council will sponsor three lectures on color by representatives of the International Printing Ink Corporation and the Massachusetts Institute of Technology. These lectures have been given before a number of organizations and have been enthusiastically acclaimed by those who have been present. They include: The Chemistry of Color, by Dr. A. E. Gessler (I. P. I.), Color as Light, by Dr. A. C. Hardy (M. I. T.) and Color in Use, by George Welp (I. P. I.)

Since these lectures have earned an excellent reputation it is possible that the attendance may exceed the capacity (400) of the Astor Gallery. Those reading this notice and secretaries of organizations interested, should notify R. G. Macdonald, Secretary of the Inter-Society Color Council, 122 E. 42nd Street, New York, as soon as possible, of their intention to be present.

If the number of reservations exceeds the capacity of the meeting room tickets will be distributed to first ones who notify the Secretary.

The A.A.T.C.C. is a member of the Inter-Society Color Council, and any member of the Association is cordially invited to attend the three lectures on color.

APPLICANTS FOR MEMBERSHIP

Active

George S. Pickles—Dyer, Goodall Worsted Co., Sanford, Maine. Sponsors: F. E. Fleming, M. P. Graham.

Elizabeth Clair Ramsay—In charge of textile work, Good Housekeeping Magazine, New York, N. Y. Sponsors: E. Freedman, W. D. Appel.

Maurice Riddiough—Dyer, Franklin Process Co., Philadelphia, Pa. Sponsors: R. W. Delaney, A. P. March.

Robert Robertson—Manager Technical Dept., National Aniline & Chem. Co., Inc., New York, N. Y. Sponsors: F. J. Allen, A. P. March.

Raymond W. Schernig—Assistant to Chemist, Wood Worsted Mills, Lawrence, Mass. Sponsors: H. D. Grimes, G. M. Thompson.

Frederick E. Sears—Chemist and Colorist, Wells & Richardson Co., Inc., Burlington, Vt. Sponsors: L. W. Craft, G. O. Linberg.

Thomas W. Shoesmith—Dyer, Goodall Worsted Co., Sanford, Maine. Sponsors: F. E. Fleming, M. J. Lane.

Murray B. Smith—Asst. Foreman Dyer, Standard-Coosa-Thatcher Co., Rossville, Ga. Sponsors: W. E. Hadley, L. A. Olney.

Charles A. Sylvester—Textile Colorist, E. I. duPont de Nemours & Co., Inc., Wilmington, Del. Sponsors: W. J. Zinck, H. C. W. Calcott.

Junior

Brittain E. Lukens Jr.—Dyestuffs Salesman in Training, E. I. duPont de Nemours & Co., Inc., Wilmington, Del. Sponsors: W. J. Zink, H. C. W. Calcott.

D. A. Siravo—Superintendent and Dyer, Horace Linton & Bros., Inc., Philadelphia, Pa. Sponsors: A. E. Raimo, A. W. Edwards.

Associate

Leo M. Morton—Salesman, Nyanza Color & Chemical Co., Ashland, Mass. Sponsors: J. N. Dalton, W. E. Lincoln.

MEETING, SOUTH CENTRAL SECTION

THE regular meeting of the South Central Section was held Saturday evening, January 24, 1936, at the Read House in Chattanooga.

Thirty members and guests were in attendance.

An informal lunch preceded the business meeting.

Chairman Harold Schroeder presided at the business meeting wherein the nominating committee made its report for officers for the coming year.

The report of the nominating committee was as follows:

Chairman—Walter E. Hadley, Standard Coosa Thatcher Co.

Vice-Chairman—Homer Whelchel, Central Franklin Process Co.

Secretary — Murray B. Smith, Standard Coosa Thatcher Co.

Treasurer — John Dabbs, Jr., E. I. du Pont de Nemours & Co.

Nominations were called for from the floor, and when none were forthcoming, the secretary was authorized to cast a single ballot for the entire slate. The above nominees were declared elected.

Mr. Pickens of the Charlotte office of the du Pont Company, addressed the meeting, presenting very acceptably a paper on silk hosiery dyeing.

In the discussion which followed the reading of this paper, the Frazer Testing Machine, which was on exhibition at the Knitting Arts Exposition, was quite freely discussed, and it was the consensus of opinion that for laboratory comparisons this apparatus possessed decided value.

Respectfully submitted,

MURRAY B. SMITH, *Secretary.*

CALENDAR OF COMING EVENTS

Meeting, Philadelphia Section, February 28, 1936, Broadwood Hotel, Philadelphia.

* * *

Meeting, New York Section, Chemists' Club, New York, N. Y., Friday, February 28, 1936.

* * *

Attention is called to the announcement of the meeting of the Inter-Society Color Council appearing on page P75 of this issue.

* * *

Annual Meeting Providence, R. I., December 4 and 5, 1936.

AMERICAN
DYESTUFF
REPORTER

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OBITUARY

GEORGE H. WHALEY

GEORGE H. WHALEY, president of John Campbell & Co. and Amalgamated Dyestuff & Chemical Works, Inc., passed away at his country home in Victory Mills, New York, on Sunday, January 19th. Funeral services were held at his home on Wednesday, January 22nd.

Mr. Whaley was well and favorably known to the members of the dyestuff industry and his loss will be keenly felt.

Announcement has been made that the business of the companies of which he was president will be conducted as heretofore.

A. W. BUTTERWORTH

A. W. BUTTERWORTH, president of H. W. Butterworth & Sons Co., passed away at his home in Langhorne, Pa., on Wednesday, December 18th. Services were conducted on Friday, December 20th.

Mr. Butterworth had been identified with the company for the past 50 years and held the office of president for the past 8 years. He was 67 years of age at the time of his death and is survived by one son, two brothers and a sister.

Book Reviews

Men, Money and Molecules, by William Haynes, Third Printing. Published by Doubleday, Doran & Co., Inc., 186 pages. Price $1.50.

An exceptionally interesting book tracing the development of the chemical industry from earliest times to the ultra-modern industry of today. Written in non-technical language the book acquaints the reader with statistical facts and figures in a manner that is far from boring. One learns, for instance, that the United States makes more chemicals—measured either in dollars or tons—than are produced in Germany, England, France, Italy, Japan and Russia all added together. In dollars this represented for 1927 a world total chemical production to the value of $5,226,000,000 with the United States standing first with $2,273,800,000; Germany second with $864,000,000 and Great Britain third with $552,000,000.

Interesting also is the chapter dealing with the manner in which man learned how to make chemicals work for him wherein we are told that many common practices were stumbled upon quite by accident. Other chapters deal with the following subjects: molecules and machines; chemicals come to America; munitions and molecules; the ultra modern industry; making chemicals; chemicals in commerce; chemical substitutes; the chemical revolution.

There are numerous photographs illustrating methods of production.

This book serves to acquaint the reader with the importance of chemicals in every-day life: the problems, responsibilities and opportunities of the chemical industry and finally the position of chemicals in the industrial picture of today. It deserves a place on every well-informed chemist's bookshelf.

Rayon and Synthetic Handbook, by E. W. K. Schwarz and H. R. Mauersberger. Second edition. Published by Rayon Publishing Corp. 559 pages plus advertising. Price $3.75.

Those who are acquainted with the first edition of this handbook will need no introduction as to the type of material which is contained in the handbook as the same material has been revised, enlarged and brought up to date. The value of the book has been increased by the addition of several entirely new chapters. In addition to the work conducted by the authors in compiling the handbook they have been assisted by various experts who are well qualified to write on some particular branch of the rayon industry.

One of the features of the new handbook is a complete and accurate history of the American rayon industry. This description embodies the first quarter century of progress and many interesting events are cited. Other chapters deal with the following subjects: economic and statistical background, by Stanley B. Hunt; raw materials and their preparation; four yarn manufacturing processes; allied rayon yarns and products; winding, soaking and throwing; warping and warp sizing; weaving of rayon goods; commercial grey goods constructions; manufacture of rayon knit goods; dyeing of rayon yarns and goods; printing of rayon and synthetic fabrics; dry finishing and special processes; testing—physical and chemical, by Irving J. Saxl; dry cleaning, by C. C. Hubbard; laundering, by G. H. Johnson; trade-marking, by O. S. Geier; yarn producers; American and foreign literature.

The book contains numerous illustrations, charts and tables.

This book should serve as a valuable reference source to all who are interested in the subject of rayon.

Washington News Letter

By WALDON FAWCETT

IT RESTS with this Congress, at this, its final session, to take a considered attitude in expression of a permanent legislative policy with respect to technical and scientific research. For upward of a decade the research situation at Washington has been in a state of flux, or has lacked definition and continuity of program. Prior to the depression, certain Departments of the Government had the benefit of fairly liberal appropriations for research. In other quarters the Federal organization was not consistently converted to research. Slashing of appropriations during the depression took toll of all Governmental research projects and institutions. During the past two years the cause of research has looked up. But the resumption or extension of activities has, in most instances, been financed by allotments from the special funds at the disposal of emergency agencies. Just in proportion as the fiscal year 1937 is counted upon for the return of normalcy, must the present Congress, in its forward planning and budgeting, attack the problem of what part of paternal or cooperative responsibility is, for the long pull, to be assigned to original research.

WINDING UP THE TEXTILE INVESTIGATION

The Federal Trade Commission is putting the finishing touches to the general investigation of the Textile Industry which was undertaken pursuant to an Executive Order of September 26, 1934. According to the original schedule the textile probe should have been completed ere this. For, in the interest of urgency, the Trade Commission was directed to give priority over other investigations to the survey of labor costs, profits and investments of companies and establishments in the textile industries in order to show what effect increased wages and other costs may have on the industry. Within two months 765 companies had reported and tabulations for a preliminary report were closed November 24, 1934. Then came the extension of span which has delayed the finals.

Under the original schedule, data covering balance sheets, income, expenses, raw and finished goods inventories, etc., was requested for three 6-month periods beginning January 1, 1933 and for a two-month period from July 1, to August 31, 1934. Then came the President's letter of January 25, 1935 with the result that the investigation was extended to cover the 6-month period ending December 31, 1934. Extra time was also required to comply with the request of the Cabinet Committee on Textiles that an addition to the Report be provided to show studies of companies grouped according to rate of return on investment, net profit on sales, and amount of textile investment. The essence of the project will, of course, be found in the deductions to be drawn from comparison of textile conditions and consequences during three successive stages, viz., pre-code era, code interim and the half-year that encompassed the derangements due to the textile strike of 1934.

PROGRESS IN FEDERAL WORK ON FAST DYES

The Chief of the Bureau of Chemistry of the U. S. Department of Agriculture reports that during the past year his Bureau has discovered a new process for doubling the yields of a whole series of ketones which are valuable in the manufacture of intermediates for dyestuffs and medicinals. Incidentally, there have been set forth the important theoretical considerations which underlie the reactions employed. These discoveries and reports are characterized as of fundamental importance in revealing the mechanism of the Friedel and Crafts reactions in organic syntheses. Furthermore, the net result is to make possible considerable monetary savings to industry through more efficient synthetic processes. The current program is the sequel to the earlier investigative and experimental work in the field of colors which resulted, notably, in the development of an economical method for the production of phthalic anhydride, to which Bureau officials ascribe much of the credit for the latter-day expansion of the American vat-dye industry.

NEW SOURCE OF STARCH FOR SIZING WARP YARN

Contact of the textile industry with the Government's effort to foster establishment of a sweet potato-starch industry in the United States was signified by the purchase by a Mississippi cotton mill for use in sizing warp yarn, of the initial run of 70 tons of starch. By this specific production, turned to practical account, the Federal movement enters a new phase. Over a period of years the Department of Agriculture has conducted surveys which have demonstrated that there is a potential market in the United States for root starches capable of replacing imported potato and cassava starches. In this explorative work the deduction was conclusive that sweetpotato starch was suitable for the sizing of warp yarns and for finishing cotton fabrics.

This, then, became the point of attack when the Federal Emergency Relief Administration vouchsafed a willingness to finance a practical demonstration. The Federal Administration allocated funds to the Mississippi Emergency Relief Administration for the manufacture of starch by the process developed by the Department. The Government provided from the laboratories at Washington the technical, chemical and engineering direction and su-

pervision necessary for the purchase, installation and operation of the equipment; conducted such plant research as was required to insure successful operation of the factory; and acted in a directing capacity in the disposal of the finished product. Actual operation of the plant was by Sweet Potato Growers, Inc. a cooperative agency organized under the Mississippi State laws for this purpose.

TRADE PRACTICE CONFERENCES AND VOLUNTARY AGREEMENTS

With the pronouncement by President Roosevelt placing finis on the record of the National Recovery Administration, as such, deepened interest will attach to the future of self government in business, via mutual compact, for fair or ethical competition. Following termination of the National Recovery Administration codes, the President, by Executive Order, delegated to the Federal Trade Commission all his authority under the National Industrial Recovery Act, as extended, to approve trade practice provisions of voluntary agreements. As a result of this move, two courses are open to industry for voluntary cooperation and agreement in establishing codes of ethics and eliminating unfair methods of competition or trade abuses. With similarity of functions and purposes in the alternative means to the common end, the choice of medium may present difficulties for some groups.

The main difference is that Voluntary Agreements under the NIRA Act as extended must contain labor provisions, whereas such provisions need not be included in a Trade Practice Conference pact. Voluntary agreements vary certain stated exemptions from the anti-trust laws. But these apply only to the labor provisions and to such trade practice provisions as do not offend under existing law. The pattern of the Trade Practice Conference is well established inasmuch as Agreements had been entered into by about 150 industries before NRA came on the scene. The rules, as shaken down, cover two general species of unfair methods of competition. In the first category are the practices which are illegal and which are due for attention by law enforcement agencies regardless of whether or not the offender has signed an agreement. In the second category are the practices which are not illegal but which the industries involved desire shall be regulated. Under the present set-up of the Federal Trade Commission the administrative responsibilities of standardization of the rules of industrial conduct fall to the Division of Trade Practice Conferences which was established in 1926. The manner in which the system is functioning may be surmised from the fact that during the past year only 34 complaints were lodged, charging violation of Trade Practice Conference Rules and of this number twenty were adjusted through correspondence.

CHALLENGE TO USE OF COAL-TAR DYES ON CITRUS FRUIT

An issue with extensive possible implications for the dyestuffs industry has been joined by the stiffening attitude at the U. S. Department of Agriculture in respect to the use of artificial coloring on fresh citrus fruit. According to Federal inspectors, the application of yellow and orange coal-tar dyes to the peel of oranges began in 1934. Almost immediately thereafter the Department warned the citrus industry that the use of artificial color must be disclosed to the consumer by a conspicuous statement on each orange and that concealment or inferiority or damage of the fruit by the added color would be in violation of the Federal Food and Drugs Act, even though the requirement for color declaration was met.

Here the matter rested for the season of 1934-35, the citrus industry in Florida meanwhile proceeding with installation of elaborate color-processing machinery. With the advent of the present shipping season, the officials at Washington made up their minds that there was violation of the law in the staining of oranges in simulation of oranges of greater maturity and flavor or of superior variety. Accordingly notice has been served upon the trade that on and after September 1, 1936, action under Section 10 of the Food and Drug Act will be taken against interstate shipments of oranges so stained and under Section 2 of that Act against the shippers on the ground that inferiority is concealed.

Users of orange dyes, who are resisting the Departmental edict, contend that the dyed fruit is mature and edible but that by reason of its uneven or unsatisfactory natural color it is regarded by the buyer as immature or inferior. Proponents of the use of coal-tar dye insist that the addition of the dye merely gives to the fruit the color associated with maturity in the minds of the purchasing public. The argument of the trade is that the requirement for a "Color Added" imprint—"plain and conspicuous"—on the skin of fruit that is not inferior, is a particular hardship since it places a stigma on the entire output in the eyes of the lay public.

From the standpoint of the dyestuffs industry perhaps the chief significance of this controversy rests in the circumstance that, in the end it may clarify the situation with respect to obscure statutes governing the use of dyes. The U. S. Food and Drug Administration is, in sentiment, opposed to the addition of chemicals of any kind to natural articles of food. However, the Food and Drug Act, as it stands, does not permit a broad prohibition upon harmless dyes if not employed to mask inferiority. The test of dye-guilt favored by Departmental officials appears to be the passing off of a color—assisted cheap product at the price of a more expensive grade. This has been the gauge employed, this past year, in extensive seizures of soybean flour colored to simulate egg noodles of a high egg content. Incidentally, the raising anew of the question of dye-privilege in the food field will be not without effect upon the ultimate language of a revised Federal Food Act, if and when the basic law is rewritten.

CHANGES IN COLOR STANDARDS

Letters of inquiry reaching Washington would seem to indicate an impression in certain quarters that the revised

(Continued on page 83)

Cellulose—

(Continued from page 60)

the pre-treatment to which the cellulose had been subjected so that the degree of depolymerization, expressed in various ways, may be well used for characterizing the various forms in which cellulose exists or into which it had been transformed[36].

The results, presented before the Bunsen Society in Dramstadt in 1925, caused *Staudinger*[37] in the discussion to direct attention to the parallelism between the long chains of polymethylenes he had synthesized from formaldehyde and cellulose (depolymerization of cellulose acetate), studies on which he had just started.

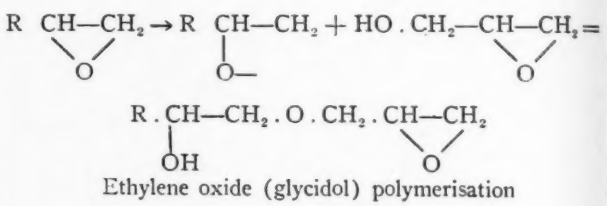
As indicated earlier, the processes of depolymerization are manifold, and it would depend upon the means applied whether they result in still rather intact or in more or less shortened chains. These processes, in some cases, may also occur when originally rather intact cellulose is forced to go into solution. On regeneration, amorphous cellulose is obtained with physical properties different from the original substance. But the structural change which might have taken place on depolymerization, either before or during the process of dissolving, has not yet become measurable, for the shortened chains are still so long that our usual means of chemical identification fail. As for the process of regeneration, i.e., the spontaneous agglomeration which frequently has been observed in cellulose solution on standing for a certain length of time, we may well be permitted to endow the shortened anhydride chains in solution with supermolecular forces which under certain circumstances may effectuate the re-association of single chains of various lengths to bundles.

But these forces are certainly not attached to the lower anhydrides which we are able chemically to identify. For quite a large number of these have to be linked together (and it is important to note that this occurs through primary valencies) before the physical properties of the thus enlarged molecule become such as are characteristic of high molecular substances, that is their colloidal nature, their lack of crystallization, their high viscosity in solution and the phenomenon of re-association. A classical example was supplied by Emil Fischer in the synthesis of larger and larger polypeptides. But not before a molecular weight of about 1200 was reached did the polypeptide behave as a colloidal substance. Later, Staudinger has, as mentioned, illustrated this principle in the long chains, the polymethylenes, which, regarding their physical properties, are well comparable with the cellulose molecule.

In the years from 1925 on *Staudinger*[37] developed his theory on the relation between molecular weight (chain length respectively) of cellulose in its various forms and derivatives and viscosity of solution. In general this theory says that viscosity in solution proportionately increases with increasing chain length and vice versa and that in solution we deal with the single chain molecules. If decreased viscosity expresses decreased chain length and presuming that dissolution does not change this length any further, it follows that on depolymerization the first step is not the breaking up of the lateral bonds between the chains but is the shortening of the chains, and the micellae remain intact until dissolution takes place. We shall return to this conception once more in another chapter of this paper.

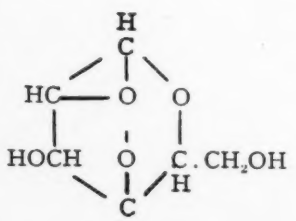
The comparability of the cellulose molecule and that of other natural products with synthetic polymers was also well illustrated on the polymeric products, amongst them polyamides and polyacetates comparable with natural silk and with cellulose, which were synthesized by *Carothers*[38] and his many cooperators (1929 and following years). On artificial threads obtained from such polymers Carothers could demonstrate that a useful degree of strength and pliability are reached only with a molecular weight of 12,000 and a length of the chain of not less than 1000 A. U.

Still more recently *Hibbert* (1933) followed similar lines[39] in building up long chains of ethylene glycol units applying the mechanism of polymerization as first established for glycidol by Nef and, for other ethylene oxide containing compounds, by Levene and Walti, for instance (*see Figs.* 9 *and* 10):



Ethylene oxide (glycidol) polymerisation

Fig. 9



Glucose Anhydride

Fig. 10

Assuming glucose to react in its ethylene oxide form and thus to be capable of undergoing polymerization in a similar way as ethylene oxide, Hibbert offered a working hypothesis for plant synthesis of sugars and polysaccharides. It is thought that to the free valence form of the ethylene oxide ring in the glucose anhydride a glucose is added to form a disaccharide. To the disaccharide another glucose is added resulting in a trisaccharide and so forth. This "intramolecular chain synthesis" is also illustrated in Fig. 11.

From the foregoing it is evident that supermolecular forces become active only when the molecule has attained a certain, larger size, and it appears as if the development of such forces, as mysterious as this phenomenon may

$C_6H_{10}O_5$	+	$C_6H_{12}O_6$	=	$C_{12}H_{22}O_{11}$
Glucose Anhydride		Glucose		Disaccharide

Fig. 11

seem, is only the natural consequence of the larger growing molecule. The primary valencies do not suffice to keep so many units tightly enough together, and assistance is needed. This is supplied by the supermolecular forces.

The fact that these forces are a function of the growth of the molecule, may be regarded as a strong support of the belief that the dispersion of cellulose in solution does not dissolve the chains into monomolecular anhydrides, nor does it seem to be justified to assume that these forces are of a latent character in the lower anhydrides. The general belief today is that cellulose in solution exists in the form of its molecules, i.e., single chains of the same length as prior to dissolving.

Accepting the conception of depolymerization, the hypothesis advanced some 25 years ago, namely that cellulose is chemically the same substance in all plants[40], would still hold true, but with the reservation that such structural changes, which result in chains still being very long, cannot be made recognizable with our present chemical tools.

Doubtlessly, the plant, our great master, builds up the high molecular saccharides, as cellulose, starch, the pentosans, inulin and so forth, according to the same principle which prevails during synthesis in our laboratories, that is to say, it uses the primary valencies of the simple compounds for synthesizing the disaccharides, the oligosaccharides and so forth, already stabilizing them by dehydration, thereafter leaving it to the supermolecular forces to endow the longer and longer growing chains with still greater stability.

It appears very plausible to identify these forces with the "van der Waalian forces of molecular cohesion," a conception to which *van Laar* first has directed attention[41]. These forces are assumed to increase with the size of the molecule. As we will see later, *Meyer* and *Mark* have attempted to estimate these forces for various chain lengths[42].

With regard to the chemical identity of cellulose in plants, there is no doubt that different plants are, and even the same plant is, capable of polymerizing single units into chains of *different* lengths, and I am quite sure that we would find this view confirmed in the differences of viscosity in solution or by other methods, if we were able to isolate the chains of various lengths without degradation.

But what we are still unable to do, some bacteria seem to do for us in the way of biochemical synthesis, for the cellulose as produced by bacterium xylinum and acetylobacter from sugars, more recently under the close and encouraging watch of *Dr. Hibbert*[43], and which needs no other treatment than washing with water to be ready for investigation is chemically the same as plant cellulose, and it also gives, as *Clark* has shown[44], and later (1933) *Champetier*[45], and *Mme. Khouvine* (1933 and 1934)[46], the same X-ray diagram as plant cellulose. It therefore should be an excellent material for enlightening us on the question of how many glucose anhydrides these bacteria are able to unite; in other words, whether or not their ability is limited compared with that of the plants.

Returning in our historical review to the year 1927 we find that it, generally speaking, had brought to light three outstanding facts: Firstly, cellulose has a high molecular weight compared with sugar anhydrides, which result from the degradation of cellulose and are monomeric. Secondly, hydrolysis renders besides glucose and cellobiose, a cellotriose, and thirdly, the linkings between the glucose units in the cellulose molecule are very likely no other than they were proven to exist in cellobiose.

Two years later (1929) *Willstaetter* and *Zechmeister*[47] produced the results they had already obtained in 1913 on hydrolysis of cellulose with strong hydrochloric acid and whereby, as mentioned before, a triose and a tetrose could be discovered. Also cellobiose was found amongst the products of hydrolysis, certainly a strong proof against the opinion that this disaccharide is not performed in cellulose but would owe its existence to a synthesis (reversion) during the milder process of acetolysis.

The polemics between representatives of the conception of the small units and those who tried to reinstate the classical conception of structural chemistry caused *Zechmeister,* some years later (1931), to investigate the hydrolysis with strong hydrochloric acid more thoroughly and he succeeded with *Toth*[48], by means of fractionated precipitation, to confirm the previous results and to add to the triose and the tetrose, a cello-hexose, although the latter not in crystallized form. Moreover, the cellotriose on acetolysis rendered octacetyl-cellobiose and is therefore to be regarded as a cellobiosido-glucose (*see Fig.* 4).

In the meantime (1930) *Freudenberg*[49] succeeded in producing the permethylated derivatives of cellobiose, cellotriose, and cellotetrose, and at the same time and during the following years in synthesizing the first two of them (the di- and tri-derivative) by allowing trimethylmethylglucoside to react on the chlorhydrines of the methylated glucose and celloboise respectively. By these syntheses, i.e., from components in which the modes of linking were known, and further by determination of the optical rotation values and finally by quantitative evaluation of the optical superposition could he prove that the two glucosido linkings in the cellobiose, the three in the cellotriose, the four in the cellotetrose, and the six in the cellohexose, all belong to the β-series. Hence it is very probable that the cellulose itself contains only linkings of the β configuration. Only one α-linking, as in maltose, to 100 β-linkings would reveal itself by a distinct change of the molecular rotation.

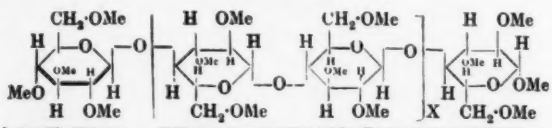
Further proof for the fact that there is only one mode of linkings between the individual glucose units of the cellulose molecule was brought forth by a study of the kinetics of the cleavage as effectuated by hydrolysis and

acetolysis. As these processes proceed with time, more and more carbonyl groups are being exposed, the number of which may be quantitatively estimated. Thus, the extent to which cleavage occurs, may be expressed as a function of time. Since the di- and oligosaccharides submit to hydrolysis more easily than the longer chains, the premise to the kinetic calculation, namely that all linkings undergo cleavage with the same ease, is not quite fulfilled. Yet, a decision could be made as to whether the linkings are equal or whether different linkings alternate.

After the fundamentals of such investigations had been established by *Meyer, Hopff* and *Mark*[50], and by *W. Kuhn*[51], *Freudenberg's* numerous investigations during recent years leave no doubt that the linkings in the cellulose are all of the same type[52].

Thus at the present time (1935) cellulose presents itself as a chain, in which numerous pairs of glucose units are linked together in a mode corresponding to that obtaining in the cellobiose, namely in 1,4 position, and only the length of the chain, i.e., the number of glucose units, remains to be discussed.

It will be remembered that the permethylated[53] cellulose on hydrolysis renders, according to *Haworth* and *Machemer*[54], besides trimethyl glucose, a certain amount of tetramethylglucose (0.6 per cent), indicating that the (non-reducing) glucose unit at the end of the chain possesses four hydroxyl groups open to methylation. The additional hydroxyl is that in No. 4 position (*see Fig.* 12).



Hydrolysis gives tetramethyl glucopyranose.

Hydrolysis gives 2 : 3 : 6-trimethyl glucose and MeOH.

(Haworth and Machemer)

Fig. 12

The proportion of tetramethyl to trimethylglucose thus furnishes a measure of the length of the chain. Haworth and Machemer have calculated it to consist of not fewer than 100 and not more than 200 glucose units, and they regard this size to be the average lower limit of the cellulose molecule. The corresponding molecular weight of the cellulose would be between 20,000 and 40,000.

This method of determining the length of the chain—apart from the fact that the latter depends upon the past history of the cellulose preparations used—may be open to criticism since the method of hydrolysis as well as that of isolation of the tetramethyl product are not quantitative. With a similar criticism we have to face the other methods which from the special significance of a terminal unit or groups try to calculate the length of the chain. This is so with *Bergmann* and *Machemer's* method based upon determination of the carbonyl group in the terminating unit[55] and, still less reliable, the method of *E. Schmidt,* who assumes a free carboxyl group[56].

Although sometimes agreement in molecular weights derived from different methods has been found, the discrepancies prevail. *Stamm,* using Svedberg's ultracentrifugal method on a cuprammonium solution of native cellulose arrived at 250 glucose units[57]. *Kraemer* and *Lansing* found higher values[58]. By measuring the breadth of the lines taken along the fiber axis in the X-ray diagram, *R. O. Herzog* and *Krüger* calculated a molecular weight of 20,000 for native Ramie cellulose[59], and similar values are found by Herzog and by Duclaux using the osmotic pressure method. Again, Staudinger, by the viscosity method, arrives at 750 glucose units. Thus, the question appears still open to discussion, unless Dr. Kraemer, who is going to enlighten us on this subject with a special paper, will be able to give us a definite answer.

In the meantime we may agree with Haworth, who says: "It is of no vital importance whether the value is to be increased to 100,000 for with either value the general structure and conformation of the cellulose are satisfactorily envisaged."

Thus, after many years of struggle and error, it was the chemical structure of the cellulose which had to be purged of fruitless speculation and to be established beyond doubt before other problems could be satisfactorily tackled.

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(To be continued)

News Letter—

(Continued from page 79)

color standards for cotton which are to become effective August 20, 1936 are a more pronounced departure from the old standards than is actually the case. Discussion of the subject may have been precipitated by the approach of the Universal Cotton Standards Conference which is to convene in March and will consider the revised standards. Specialists insist that the modifications which were duly announced last August are no more than fine justification in their color changes of the crop during the past few years,—a consistent trend to White cotton and away from the creamy or "buttery" color. In deference to the color realignment of the crop as a whole, the new standards, based on careful color analysis, so affect the Yellow Tinged and Stained grades that the Tinged grades no longer carry so deep a color as formerly whereas the Yellow Stained grades cover all cottons that are deeper in color than Tinged.

WILL WEBB LAW EXPORT REVIVE?

Negotiations by the U. S. Department of State of a series of reciprocal Trade Agreements will precipitate, for many interests in the textile—chemical fields the question of whether and to what extent Webb law associations will again be summoned to facilitate export trade. During the depression, manufacturers in certain lines reported that they would be unable to export without Webb law organization. On the other hand, price demoralization in various foreign markets has caused some members of Webb Act groups to be unwilling to ship. Hence some of the associations suspended price agreements, permitting members to sell at independent prices. Now comes the question of the effect of the changing status of future policies. Agreements upon prices have not been the only objectives in the get-together movement legalized by the Webb Act. Important also have been the exploration of new markets; joint advertising and promotion of members' products; consolidation of shipments, etc.

● DIRECTOR, TECHNICAL SERVICE

Norman A. Shepard has been appointed director of technical service for the American Cyanamid Company, the American Cyanamid & Chemical Corporation and other companies in the Cyanamid group where cooperation in this field is required. He will be responsible for directing the technical service laboratories located in Stamford, Connecticut, the expansion of the sales service laboratories to meet present and increasing need and the coordination of the technical and sales service work in all of the several divisions of the above named companies.

Dr. Shepard is a graduate of Yale University (Ph.D. 1913), was Assistant Professor of Chemistry at Yale University from 1917 to 1919 and has been director of the research laboratories of the Firestone Tire and Rubber Company from 1919 to date. He will begin his work with the Cyanamid group on or about February 15th and will have his headquarters at the Stamford Laboratory.

● S. C. I. MEETING

The Society of Chemical Industry, American Section, will hold a meeting on February 21, 1936 at 7:45 P.M. at The Chemists' Club, 52 East 41st Street, New York City. Robert J. Moore will preside over the meeting at which E. R. Bridgewater, manager of the Rubber Chemicals Division of E. I. du Pont de Nemours & Co., is to be the guest speaker.

Open Forum

See previous issues for details of this department.

Answers

31—Occasionally we experience difficulty with worsted and zephyr knitting yarns, in the dyeing of colors such as dark browns and similar shades. It seems that some of the skeins come up in an entirely different shade than others.

Upon checking after dyeing, we find that the darker skeins still contain oil, and after rescouring the dyed yarn, we manage to get a uniform shade. Our method of scouring is as follows:

In a 500-pound Klauder-Weldon dyeing machine, we use about 25 pounds of olive oil soap dissolved. (Weight of soap before dissolved.) Forty pounds of soda ash light 58 per cent, 10 pounds of ammonia, temperature from 100° to 120° F. Rinsing in 120° to 130° F.

This may all be due, of course, to the fact that yarn is steam-conditioned at the mill before it comes to us, and the oil becomes so set that it does not wash out freely.

Perhaps some of your readers can suggest a scouring formula different to that we are using, which will result in more uniform dyeings. Perhaps they may also be able to explain why such a variation occurs.—**D. Y. B.**

ANSWER No. 3—During the past few years, considerable experimental work has been conducted on the development of a stock lubricant which is completely soluble in water, not emulsified; is not precipitated by acid dyeing solutions; is non-oxidizing; non-corrosive; practically colorless and odorless after application; and possesses the fiber softening and lubricating powers essential to satisfactory carding, combing, and spinning.

Such a lubricant, containing no oil, is now available Stock, yarn, and cloth containing it, may be dyed uniformly to bright shades without a scouring previous to dyeing. The lubricant washes out of the material during dyeing by virtue of the action of the dye solution passing through the goods, or by the goods passing through the liquor. The lubricant remains dissolved (not just emulsified) in the solution without precipitation or scum which would cause resists.

This water soluble lubricant eliminates the time and expense involved in scouring goods prior to dyeing, and eliminates the presence of residual oil and soap after scouring. By so doing, economies are realized, and one of the major causes of faulty dyeing is removed.

Treatments with steam to set twist and to condition yarns are not always uniform. Those portions of the yarn receiving more severe treatment will dye a different shade because of the greater action on the oil lubricant. The lubricant is not as readily removed either due to a greater degree of oxidation, or because it has been more firmly secured to the fibers chemically and physically. Such treatments with steam do not adversely affect the water soluble lubricant. It is readily removed, or may still be left in the goods during the dyeing operation. If further information is desired in regard to such a soluble lubricant please write to the editor.—NDH.

● GENERAL RELEASES

General Dyestuff Corp. announces the following releases:

Diazo Rubine B H—which produces full bordeaux shades when diazotized and developed with beta naphtol. It is stated that it can be discharged to a pure white and that it possesses very good fastness to washing. Circular G.D.C.-120.

Eulan B L—a new Eulan brand of I. G. manufacture, offered to the trade by General. It is said to be soluble in organic solvents and especially recommended by the manufacturers for use in dry cleaning. It is applied in a 10% solution which is said to produce a safe protection against moths. It is further said to be of special interest for mothproofing finished goods, worn articles, or any goods sensitive to treatment with water. Circular I.G.-1088.

Fastusol Yellow L R T A Extra Conc.—a direct dyestuff said to be of very good fastness to light which produces a reddish yellow on rayon and cotton, and is also well suited for union dyeing. It is said to leave acetate silk white. Circular G-142.

● CIBA SOUTHERN OFFICE

Ciba Company, Inc. announces the closing of its Greensboro, N. C. office and the opening of a new branch office located at 1517-23 Hutchison Ave., Charlotte, N. C. The office was officially opened on February 1st.

CLASSIFIED

The rate for "Position Wanted" advertisements in this column is 2 cents a word—with a minimum of 50 cents per insertion. For all other types of advertisements—i.e., help wanted, machinery or supplies for sale—the rate is $5.00 per column inch or less per insertion.

FOR SALE—Two Smith-Drum Hoist-Type Dyeing Machines, Monel Metal Tanks, one 5 and one 10 Pyrex Spindles. Operating now and in perfect condition. Low Price. Reply Box No. 889, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

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